

EROSION BEHAVIOUR OF 13Cr-4Ni MARTENSITIC STAINLESS STEEL

A DISSERTATION

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CANDIDATE'S DECLARATION

I hereby certify that the work which is being presented in the dissertation, entitled, “**EROSION BEHAVIOUR OF 13Cr-4Ni MARTENSITIC STAINLESS STEEL**”, submitted to the **Metallurgical And Materials Engineering, Indian Institute of Technology, Roorkee, India**, in partial fulfilment of the requirements for the award of the Degree of **Master of Technology** in “**Metallurgical And Materials Engineering**” is an authentic record of the work carried out by me during the period July 2017 to May 2018, under the supervision of **SHRI SHARVAN KUMAR**, Assistant Professor, Metallurgical And Materials Engineering , Indian Institute of Technology, Roorkee, India.

The matter presented in this thesis report has not been submitted by me for the award of any other degree of this institute or any other institutes.

Date: 15/05/2018

Place: Roorkee

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CERTIFICATE

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

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Date: 15/05/2018

Place: Roorkee

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ABSTRACT

The studies and investigation are being done on the effect of several heat treatments tested on the structure. The erosion behaviour of 13/4 martensitic stainless steel that is CA-NM and its mechanical properties were also been investigated. Nominal composition in weight% is as follows:

C-0.020 Mn-0.18 Si-0.14 Cr-13.75 Ni- 3.45 P-0.018 S-0.009

In case of hydroelectric power projects, this steel is greatly used for the fabrication of underwater parts of such project. A thorough investigation has been conducted in order to develop resistance of high erosion by giving several heat treatments to the cast steel.

Various heat treatments were given to the as received bars of the 13/4 martensitic stainless steel. Austenitization of cast steel at the temperatures ranging 950°C, 1000°C and 1050°C along with the holding time of 2 hours, 4 hours and 6 hours respectively are given under heat treatments. At the temperature of 600°C, the tempering is being done for an hour followed with quenching of oil.

The treated steel specimens is tested for Tensile strength (UTS), Toughness (Impact Strength), Hardness, and Ductility (% Elongation) and their effect on erosion behaviour.

Optical microscope is used for identifying carbides, lath martensite or the similar other distinct micro constituents in the as received or the steel being heat treated steel. It is studied by using the optical microscope. The micro constituents precipitates during the heat treatment along the boundaries of grains.

Scanning electron microscope (SEM) is used for investigating the mechanisms of failure of material in tensile test, impact test and erosion test. From the impact of particles of silt, the plastic deformation occurs at the target surface during the first stage of erosion. Ploughs and material lips are formed due to this. Due to the shear, the lips are produced that later on becomes brittle. This brittleness causes the removal of material from the surface. Subsequently, a rough pattern is caused on the target surface as the material gets eroded frequently. Cutting mechanism is also vulnerable during erosion. Further for identifying the mechanism of erosion, the eroded surface is scanned under SEM.

The erosion behaviour is also affected by the microstructural aspects. For the resistance from high erosion, tempered martensite is the best option. The decrement in erosion resistance

is result of the coarsening of carbides. The studies done on SEM reveals that the erosion occurs by cutting and ploughing trailed by the formation of lip.

Several mechanical properties and the microstructure affects the erosion behaviour. It is also observed from the studies that the erosion is also likely to get affected due to the inclination of the material with respect to particles of silt present in the slurry. The resistance of erosion is improved with the increase in the toughness and ductility. UTS as well hardness also impacts the behaviour of erosion in the way such that the weight loss increases with the increase in UTS. The same goes with the hardness.

The minimum weight loss in erosion nears to 950°C-2hr-OQ-600°C-1hr. As compared to the as received material, around 36% loss in weight is observed.



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INTRODUCTION

Energy plays an important role in the economic and social development of a nation. For developing country such as India, there is great importance of secure and sustainable energy at micro level. There are different sources of energy categorised into conventional and Non-conventional energy resources. Thermal, hydro, nuclear are conventional sources whereas wind, solar or biomass are non-conventional energy resources. There is a need to develop hydropower potential as it is a renewable source of energy which do not pollutes or harms the environment. However, only 17% of the hydropower potential has been harnessed till yet. Moreover, it possesses 26% of the share in total installed capacity of India which is 126,089 MW including all the resources which is less than an ideal hydrothermal mix of 40:60.

Turbines in small hydropower plants gets damaged due to various reasons and their performance get severely declined, therefore there is a need of proper management of these power plants to achieve enhanced efficiency of hydro turbines. The damage in these turbines could be caused due to erosive wear in them affected by high intensity of content of abrasive material in monsoon. Most of the small hydropower plants were situated in hilly and steep terrains and are run-of-river schemes, hence this problem is certain to be caused. A large amount of sediments (around 20,000 ppm) is present in water during rainy season, hence making it troublesome in removing all the sediments before it passes through the turbine.

Silt is the major cause of damage to turbine. It possesses high quantity of quartz (around 70-98%) being extremely hard (hardness 7 in Moh's scale) leading severe damage to turbine components and water passage components such as guide vane, top and bottom ring liners, runner blades, labyrinths etc. Alteration of blade profile, fatigue damage or increased vibration were due to erosion wear in turbines.

13Cr-4Ni steels possess excellent mechanical properties and corrosion resistance thereby making them suitable to be used in hydro turbines and water pumps. However, these materials were less resistant to erosive wear.

These easily gets damaged due to excessive solid content entrained in the water. Forced outages, extensive repairs and drop in efficiency are all results of erosive wear damage caused

by silt. The reports reveal that hydropower stations bears a loss of \$120-150 million every year due to silt erosion.

The great amount of damage to the components is caused by Slurry erosion. The hard particles striking the surface being carried by a gas stream or entrained in the flowing liquid causes wear named as Slurry erosion. The erosion damage in conventional pipelines is very inconvenient and costly.

In the year 2000, the Department of Trade and industry in UK, had to bear approximately £20 million per annum as erosion costs alongwith 1.5% of GNP was invested as jots wear.

Conventional turbine blades were made up of low carbon steel, stainless steel, low manganese steel, white cast iron or plastic resin results in very low erosion resistance. The blades of these turbines get easily damaged with or without sediments or solid particles under high speed water impingement and thus, it interrupts hydraulic power generation. The above-mentioned cases reveal an important need of developing more erosion resistant material. Nitronic 60 steel, Nitronic 50 steel, martensitic steel etc. are some of the materials possessing more resistance properties.

Tin coating is a wear resistance coating. It is one of another effective way to reduce erosive wear caused due to slurry. The surface of the component is modified suitably through different hardening process such as pulse plasma nitride and laser hardening for counteracting the silt erosion problems.

LITERATURE REVIEW

2.1 INTRODUCTION

Martensitic stainless steel and austenitic stainless steel were the most widely used steels for thermal and hydro power plants. Presently, martensitic stainless steel is excessively used steel for the manufacture of underwater parts of hydroelectric projects and the such similar applications. The improvement over both the stainless steels (martensitic and austenitic steel) has led to the precipitation hardening stainless steel. However, martensitic steel had become the most widely used steel for the hydro turbines at the global level.

The family of 13 pct. Chromium steel (CA-6 NM as cast designation) popularly known as 13/4 Cr-Ni stainless steel of martensitic grade need not be specifically introduced. There were vast applications of this steel.

It possesses various qualities such as better toughness, high yield, ability to resist corrosion and erosion, fatigue strength alongwith resistance to cavitation. The package of all these qualities in a single material have increased the importance of 13/4 stainless steel due to which it has become the explicit need in the field of worldwide water turbine construction. The typical composition of martensitic steel is clarified below in weight percentage.

Table 2.1 Typical composition of martensitic stainless steel

C	Mn	Si	Cr	Ni	P	S	Mo	Fe
0.06	1.0	1.0	11.5-11.0	3.5-4.5	0.04	0.04	0.04-1.0	Balance
0.15 max	1.0	-	11.5-13	-	-	-	-	Balance
0.20 max	1.0	-	15-17	1.2-2.5	-	-	-	Balance
0.60-0.75	1.0	-	16-18	-	-	-	0.75% max	Balance
0.75-0.90	1.0	-	16-18	-	-	-	0.75% max	Balance
0.95-1.20	1.0	-		-	-	-	0.75% max	Balance

CA-6 NM is an alloy normally used in the tempered and normalised condition in which the microstructure is essential to be 100 pct. Martensitic. An adequate amount of retained austenite is contained by CA-6NM as the optimum combination of ductility, strength, toughness and hardness has been provided by this structure. For enhancement of one or more of these properties, the variation in heat treatment shall be done.

The increasing severity of requirements of Fatigue strength, brittle fracture and resistance to erosion in hydraulic power station construction and also in the field of nuclear reactors has firmly increased the use of 13/4 chromium steel with better ductility, improved weldability and higher strength.

2.2 CA-6 NM (13/4) STAINLESS STEEL

The CA-6NM named 13/4 Cr-Ni is an Fe-Cr-Ni-Mo alloy of low carbon content which is hardenable by heat treatment. Ferritizing effect of low carbon content is being offset with the addition of nickel to the composition such that strength and hardness properties are comparable to CA-15 (wrought grade 410) and the impact strength is about twice as high as is the resistance to damage from erosion or cavitation effects the addition of molybdenum confers the increased resistance on alloy to seawater corrosion. CA-6NM are castes with heavy sections and complex structures with less difficulty other than those experienced with CA-15 alloy.

The typical composition of CA-6NM stainless steel is given in the below mentioned table:

Composition pct. Max.								
C	Mn	Si	Cr	Ni	P	S	Mo	Fe
0.06	1.00	1.00	11.5-14.0	3.5-4.5	0.04	0.04	0.4-1.0	Balance

Cast : ASTM-A743, A487, A351(CA-6NM), A757(E3N)

Wrought : None

Commercial name : 13/4 Cr-Ni (Non - standard)

2.3 CONSTITUTION OF MARTENSITIC STAINLESS STEEL

As shown in figure 2.1, an austenite field in Fe-Cr constitution diagram is the basic necessity for a martensitic stainless steel. Crystal (austenite) range constituted by the chromium with increasing alloying content until it disappears completely from approximately 12% Cr onwards. It clarifies that the alloys with more than 12% Cr do not show any γ to α transformation, and thereby ruling out any accompanying grain refinement and possibility of steel hardening. In the temperature range between 1400°C and 1100°C, with the chromium content below 12%, a transformation of primary to crystals takes place.

Most often in combination with a grain refinement along with formation of martensitic structure, the austenite is transformed back into α iron at the temperature below 900°C. Due to high Cr content, embrittlement can occur. Also, with higher contents ($\approx 45\%$) the brittle σ phase starts to precipitate from ferrite at about 820°C.

Immediately after the end of solidification, iron-chromium-nickel alloys consist of the following phases:

1. Primary (δ) ferrite
2. Primary (γ) austenite
3. A mixture of ($\delta+\gamma+L$)

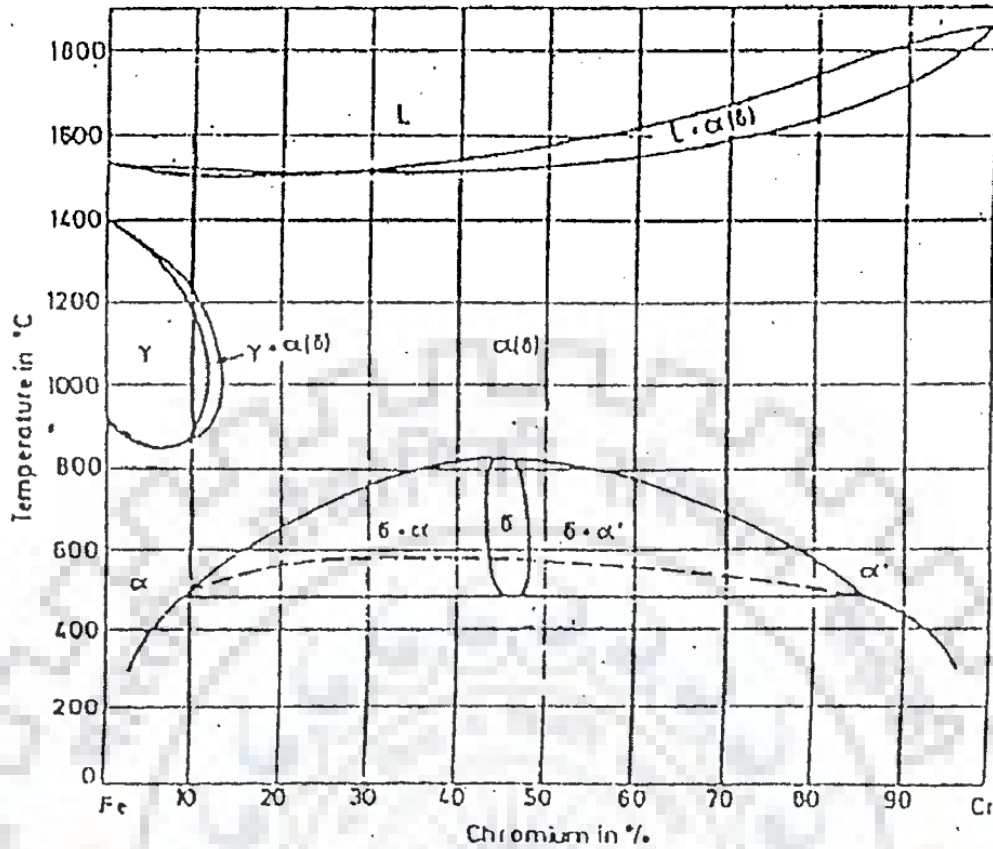


Fig. 2.1 Fe-Cr constitution Diagram

As shown in the above-mentioned diagram, the phenomenon of the ternary iron – chromium- nickel constitution in the form of two isothermal profiles at 1400° C and 1100° C. At 1400° C, the area of primary precipitated σ and crystals are gathered from the profile. In the iron corner, in Between the area of already solidified $\delta(\alpha) + \gamma$ mixture which, with nickel and higher chromium, passes into the three-phase region of $\delta(\alpha) + \gamma + L$ and finally into still liquid melt.

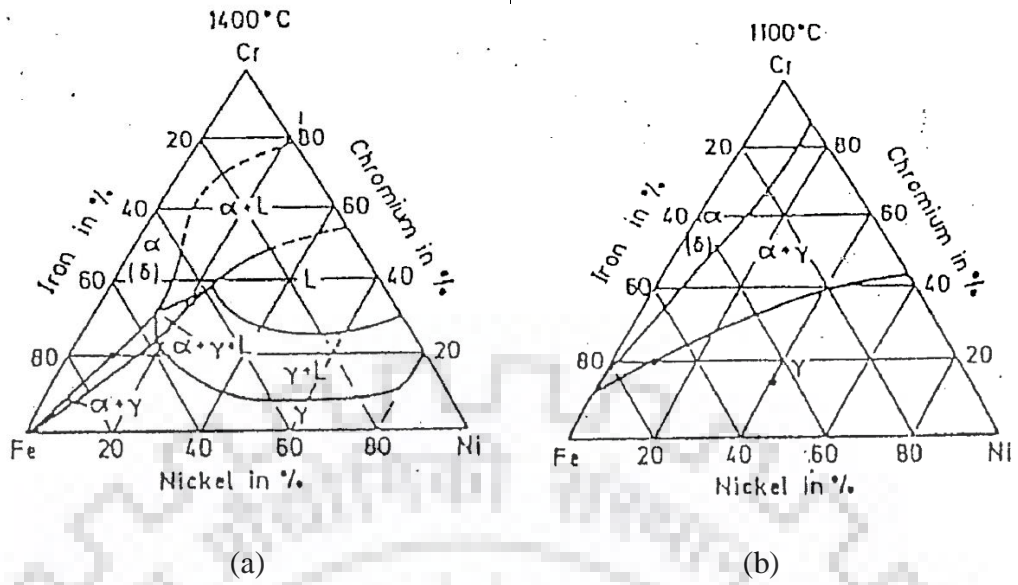


Fig.2.2 Isothermal Profile of Ternary Fe-Cr-Ni Constitution Diagram

(a) at 1400°C and (b) at 1100°C

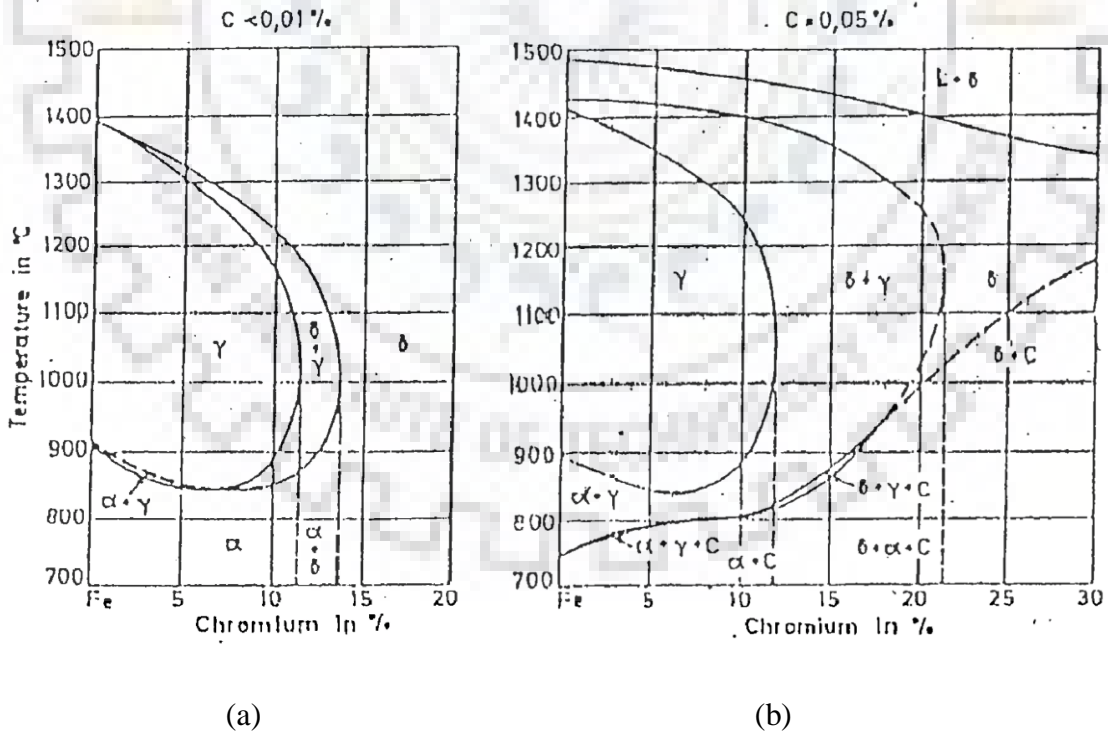


Fig. 2.3 Ranges of and Phase in the Fe-Cr constitution Diagram

In the above-mentioned figure, at 1100°C in the isothermal profile shows that the has greatly expanded. A ferrous alloy with 20% Cr, 10% Ni, 70% Fe has been plotted as a specially marked dot in the both the profiles at fig. 2.3. at 1400° C, it is apparent that it solidifies from the melt to primary phase which consists however at 1100°C of secondary formed phase. This means that primary precipitated ferrite is transformed during cooling from 1400°C to 1100°C into secondary austenite. This transformation could be partial or complete depending upon the steel grade and cooling rate.

2.4 HEAT TREATMENT OF MARTENSITIC STAINLESS STEEL

By heating to the austenitizing range of 925°C to 1065°C alongwith cooling in air or oil, martensitic stainless steel gets hardened easily.

Stainless steel's thermal conductivity is characteristically lower than that of carbon and alloy steels. Warpage and cracking in some parts are caused due to high thermal gradients and high stresses during rapid heating. Therefore, accordingly, for avoiding the above-mentioned problems, preheating is usually recommended in the treatment of martensitic stainless steel.

At the temperature between 550 C to 650C, preheating is generally accomplished. Also, heating needs to be continued only long enough that all portions have reached the preheating temperature.

2.4.1 QUENCHING

The martensitic steel can be quenched either in oil or air because of their high hardenability. In these grades, some decrease in corrosion ductility and resistance may occur due to air quenching. Through the temperature range of about 870°C to 540°C, if heavy sections are cooled slowly, these steels may precipitate carbides along the grain boundary areas. These alloys may impair their resistance to corrosion very slow cooling rate in bright annealing. For the prevention from distortion or quench cracking, air cooling is preferred and required for large and complex sections. Oil quenching is preferred.

From a temperature of 950° C to 1065° C, the castings of the CA-6 NM compositions should be hardened by air cooling or oil quenching.

While cooling from elevated temperatures, CA-6 NM is not prone to cracking. Through the choice of tempering temperature, a wide choice of mechanical properties is available.

At the temperature between 595°C to 620°C, castings of CA-6 NM is generally normalised, supplied and tempered.

The amount of Reaustenitizing increases with increasing temperatures. Reaustenitizing generally occurs upon tempering above 620° C. Cooling from such tempering temperature may adversely affect toughness and ductility both through the transformation to untampered martensite. This all depends on the amount of the transformation.

The amount of minimum reach is significantly high, even though the alloy is characterized by a decrease in impact strength in the case when tempered in the range of temperature between 370° C to 595° C. With the presence of molybdenum and nickel in the composition alongwith lower carbon content, there is an improvement in impact toughness. When the alloy is tempered above 510° C, the best combination of toughness alongwith strength is obtained.

2.5 WEAR

The study of the part of the discipline of tribology includes the processes of wear. The definition of wear includes the damage of one or both surfaces such that it generally involves progressive loss of material.

Wear is more clearly defined as a process where interaction of surfaces or bounding faces of a solid with the working environment results in the dimensional loss of the solid, with or without the loss of material. Loads (types include unidirectional sliding, reciprocating, rolling, impact); speed, temperature, types of contact and counterbodies (solid, liquid, gas) are all the parts of Wear environment. The type of contact could be (single phase or multiphase in which phases involved can be liquid plus solid particle plus gas bubbles).

The loss of material during wear was expressed in terms of volume in the Standard wear test. The standard wear test is for example, those formulated by respective subcommittees under ASTM Committee G-2. At the time of comparing wear resistance properties of materials with large variations in density, the volume loss particularly gives a truer picture than weight loss.

Wear causes creep, fracture toughness and fatigue alongwith other aging processes that causes progressive degradation of materials with time leading to failure of material at an

advanced age. The property changes during usage under normal operating parameters into three different stages mentioned as follows: -

- a) Primary or early stage or run in period. In this rate of changes can be high.
- b) Secondary or mid age process where a steady rate of aging process is maintained. The most of the life span of this age is useful or working
- c) Tertiary or old age stage. In this stage, rapid rate of aging leads to early failure.

Stress, strain rates, high temperatures or sliding velocities are some the severity of environmental conditions that results in the shortening of secondary stage and merging of primary stage with tertiary stage. These all results in reducing the working life. To minimize wear and extend working life of material, Surface engineering processes are used.

There are four principle wear process mentioned below:

1. Abrasive wear
2. Adhesive wear
3. Erosive wear
4. Corrosive wear.

2.5.1 ABRASIVE WEAR

The abrasive wear mechanism works in the way the processes we use for shaping the materials. Machining, grinding, lapping or polishing were all included in abrasive wear mechanism. When the one surface (being harder than the other) cuts material away from the second, this results in two body abrasive wear. However, this mechanism changes to three body abrasion wear very often as the wear debris then acts as an abrasive between the two surfaces. Abrasives can act as in grinding where the abrasive is fixed relative to one surface or can be as in lapping where the abrasive tumbles producing a series of indentations being opposed as a scratch.

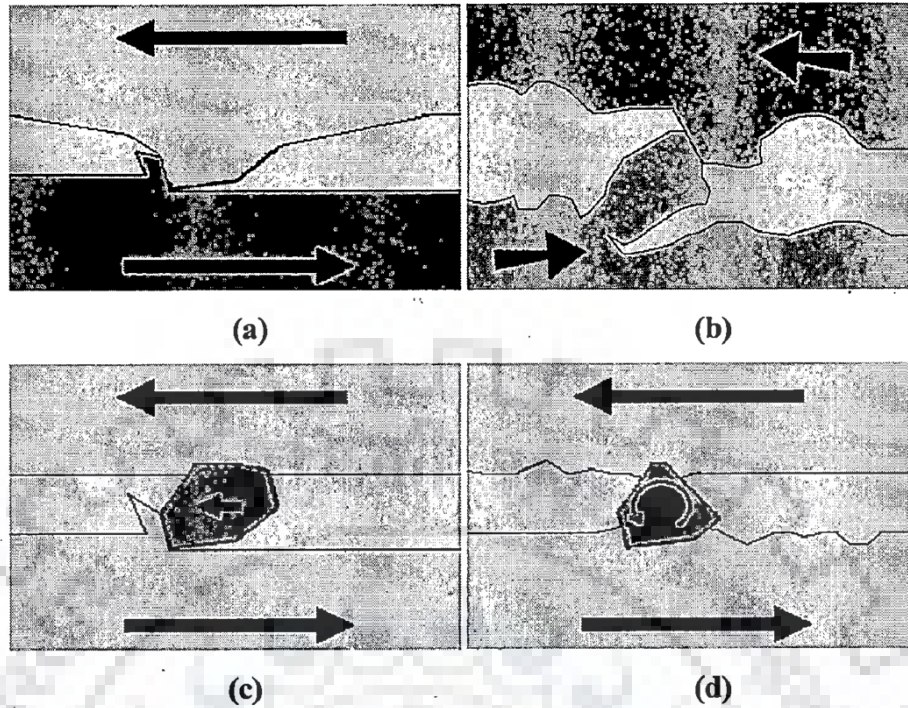


Fig 2.4 Schematic showing abrasion due to (a) hard surface (tool) cutting softer material during machining, (b) surface irregularities, (c) a rough, hard surface or a surface mounted with abrasive grits sliding on a softer surface, and (d) free abrasive grits caught between the surface with atleast one of the surface softer than the abrasive grits.

2.5.2 ADHESIVE WEAR

Scoring, seizing or Galling are some the alternative names of adhesive wear. When two solid surface slides over one another under pressure, adhesive wear tends to occur.

Asperities and surface projections are deformed plastically and welded together by the pressure of high local. These bonds are broken producing cavities on the surface when sliding continues. Also, along with it produces projections on the second surface and frequent tiny, abrasive particles, all of which contributes to the future of wear of surface.

Subsequent shearing of welded junctions between two sliding surfaces tends to the formation and production of adhesive wear. It is necessary for the surfaces to be in intimate contact with each other. The tendency to reduce the occurrence of adhesion is reduced by the surfaces which are held apart by lubricating films or oxide films etc.

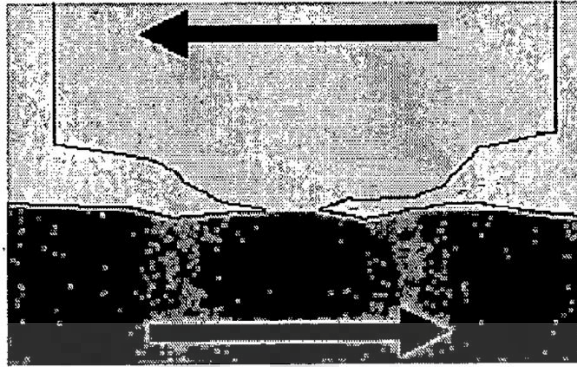


Fig 2.5 Schematic showing adhesive wear of the sliding surface during shearing of an interface

2.5.3 EROSIVE WEAR

Erosive wear is defined as the wear caused by the hard particles striking the surface, either carried by a gas stream or entrained in the flowing liquid.

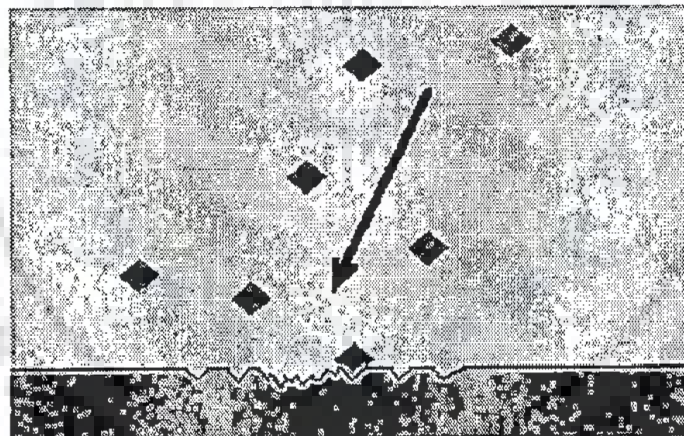


Fig 2.6 Schematic showing erosive wear of the sliding surface during shearing of an interface

This type of wear is called erosion, often qualified as solid particle erosion or solid impingement erosion. It distinguishes the damage caused by the impact liquid jet or drops.

However, the term can be applied outside the range as defined. The range in particle velocities in the erosive wear are generally between 5 and 500 m/s.

2.5.4 CORROSIVE WEAR

Corrosive wear is often referred to simply as “corrosion”. The deterioration of useful properties in a material due to reactions with its environment is termed as Corrosive wear. The formation of compacted oxide layer glazes which under certain circumstances reduces wear is formed under high temperature corrosive (oxidative).

2.6 EROSION

The solid surface gradually gets worn by the action of fluids and particles, this situation is termed as Erosion. Under four different conditions, erosion of material can occur

- (i) Impingement of solid particles against a solid surface.
- (ii) Impingement of liquid droplets against a solid surface.
- (iii) Flow of hot gases over a solid surface, and
- (iv) Cavitation at a solid surface.

This is clarified from the above facts that in erosive wear, the erosion behaviour is sensitive function of the ductility of the surface, the liquid media that transports the solid particles and the impingement angles. The factors responsible to delamination is very similar to the basic mechanism of erosion [8]. When two materials slide against each other, the wear volume V is linearly proportional to the slide distance S and normal load L but is inversely proportional to the hardness H of the material.

This may be expressed as:

$$V \propto LS/H \quad \dots(2.1)$$

$$V = KLS/3H \quad \text{or} \quad K = 3VH/LS \quad \dots(2.2)$$

In the above equation, K is dimensionless proportionality constant commonly known as “wear coefficient”. In the equation 2.2, the factor 3 is the result of Archard model. All the erosion mechanisms depend greatly on the mechanical behaviour of solids except erosion due to hot gases.

Before reviewing the phenomenological aspects of erosion, the dimensionless erosion rate (E) is defined as:

$$\text{Erosion rate (E)} = \frac{\text{weight removed from target surface}}{\text{Total weight of impacting particals}}$$

Erosion is being classified by Bhushan [11] under the different categories:

1. Cavitation erosion
2. Solid particle erosion
3. Liquid impingement erosion.

2.6.1 CAVITATION EROSION

The formation and collapse of, within a liquid of cavities and bubbles that contains vapour or gas is known as Cavitation. Generally, the cavitation is originated from the changes in pressure in the liquid brought about by the turbulent flow or by vibration but can also occur from changes in temperature(boiling).

When bubbles or cavities collapse on or very near to the eroded surface, cavitation erosion generally occurs. Liquid impingement erosion causing direct localised damage of the surface or by inducing fatigue is similar to the mechanical shock induced by cavitation.

For the better illustration of this, a picture of the localized cavitation damage on the blade of the mixed flow pump, fabricated from an aluminium -based alloy, is induced in the figure2.7

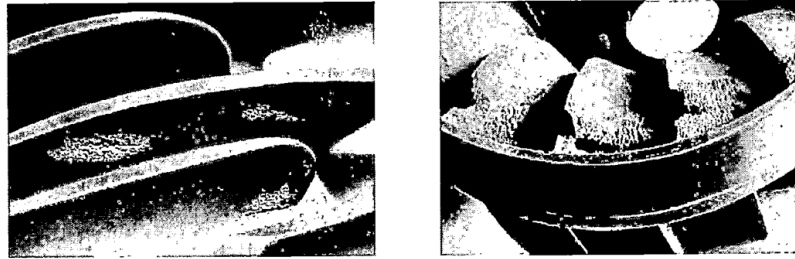


Fig2.7 photograph of the localized cavitation damage on the (a) blade of a mixed flow pump impeller made from an aluminium based alloy (b) blades at the discharge from a Francis turbine.

In the figure 2.7(b), the more extensive damage is illustrated which shows the blades at discharge from a Francis turbine. In the much larger scale flows, the cavitation damage can also occur. Reaction of the material of the solid boundary to the repetitive shock (or water hammering) loading is the other important facet of the cavitation damage phenomenon. There are various measures of the resistance of particular materials to cavitation damage have been proposed.

2.6.2 SOLID PARTICLE EROSION

When on the surface of the material, there is a loss of material by the repeated impact of small solid particles, this is termed as the “Solid particle Erosion”. Solid particle erosion (SPE) is expected whenever hard particles are entrained in a gas or liquid medium, impinging on a solid at any significant velocity. Due to their deceleration in erosion, the force is exerted by the particle on the material. Commonly, erosion involves the velocity of impingement upto 600m/s and particle sizes of up to 1000 μ .

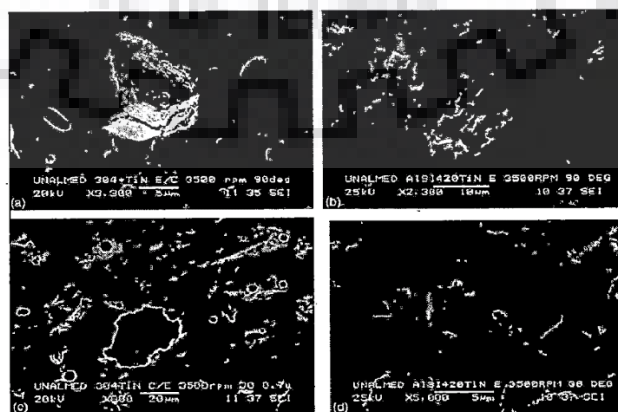


Fig 2.8 Wear marks after slurry erosion AISI 304 and AISI 420 steel [12]

The above-mentioned pictures clarify that this type of wear is called Erosion, often qualified as solid particle erosion or solid impingement erosion to distinguish it from the damage caused by the impact of the liquid drops or jet. Also, the wear is termed as the Slurry erosion if the hard particles are carried by a liquid.

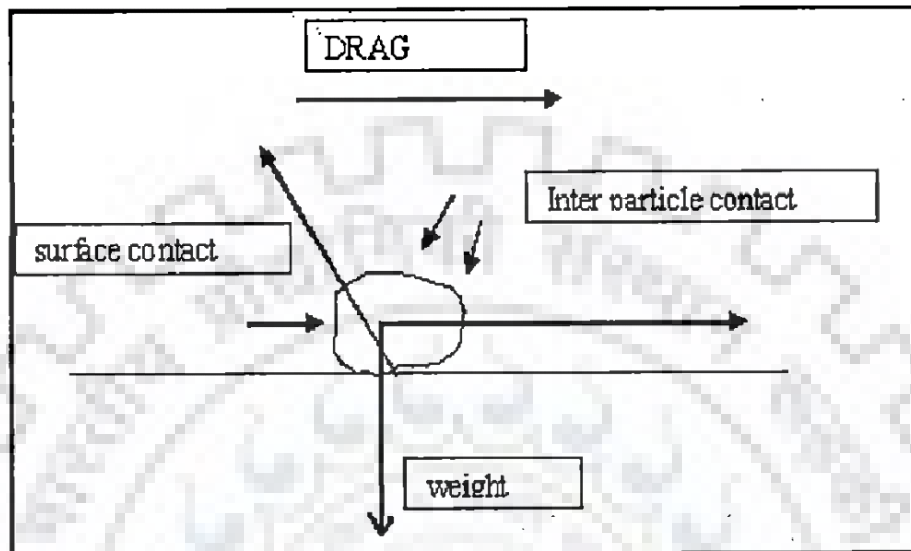


Fig 2.9 Forces acting on a particle in contact with solid surface [4]

The above diagram i.e.; fig.2.9 demonstrates that in erosion, several forces of different origins are there which may act on a particle in contact with a solid surface. The neighbouring particles may exert contact forces and however if a flowing fluid, if present, shall cause drag. On an erosive particle, the dominant force which is mainly responsible for decelerating it from its initial impact velocity, is usually the contact force exerted by the surface. In the case of abrasive wear, depending on the normal load pressing the particle against the surface on the distance slid, the amount of material is removed accordingly. In the erosion, the extent of the wear depends on their impact velocity instead on the numbers and the mass of the individual particles striking the surface.

Plastic deformation and brittle fracture are both involved in the erosive wear. Plastic flow is usually involved in the erosion of the metals, whereas more brittle materials may wear predominantly either by flow or by fracture which greatly depends on the impact conditions.

2.6.3 LIQUID IMPINGEMENT EROSION

When the surface of a solid is stroked by the small drops of liquid at high speeds (as low as 300m/s), then very high pressures are experienced, exceeding the yield strength of the most materials. Thus, the single impact is obtained from fracture or plastic deformation and the pitting and the erosive wear were lead from the repeated impact.

2.7 MECHANISM OF EROSION

Many research and studies has been done to study and understand the mechanism of erosion by solid particles [8]. There are two types of common mechanisms of particle erosion. One is the “abrasive erosion” and the second one is the “impingement erosion”.

The micro-machining action of the particles is the abrasive erosion in which it impacts the material surface at small angles whereas at large incident angles, impingement erosion tends to occur.

2.7.1 ABRASIVE EROSION MECHANISM

It is not worthy to associate abrasion with erosion because the damages due to erosive particles are often referred to as “abrasive erosion”. This is a low stress abrasive process where impact does not generate sufficient stress to fracture abrasive. This process is generally termed as “micro-machining” or “scratching abrasion”. An analytical mode was developed by Finnie [13] for predicting erosion rates that were based on the assumption that the mechanism of erosion was that of micro-machining. An equation of motion of the particle tip was the basis of the model.

There are assumptions to the model such that it assumes that a hard, angular particle, impinging upon a smooth surface with velocity V at an angle attack α , much like a sharp tool shall cut into the surface. The volume removed per impact equals the crater volume generated by the cutting particles is also further assumed by the aforesaid model. Finnie expressed the erosion rate E due to the total mass M of abrasive particles as follows:

$$E = [\rho / P\Psi] [MV^2 / J] (\sin 2\alpha - 6/J \sin^2\alpha) \quad \text{when } \tan\alpha \leq J/6 \quad \dots (2.3)$$

$$E = [\rho / P\Psi] [MV^2] J \cos^2\alpha / 6 \quad \text{when } \tan\alpha \geq J/6 \quad \dots (2.4)$$

Where

ρ = surface density

p = constant component of contact stress

$\Psi = 1/y$

l = grain contact surface length

y = depth of cut

J = ratio of vertical to horizontal force components

According to this model, maximum erosion occurs at $\tan 2\alpha = J/3$

2.7.2 IMPINGEMENT EROSION MECHANISM

This is the second damage of mechanism where at the large incident angles, impact of the abrasive particles occurs. By contrast, repeated indentations and extensive surface roughening by plastic deformation tends to be involved at the high angle erosion. This process produces extrusion forging of the material being vulnerable for the removal by subsequent particles and finally initiates the mechanism of the platelets.

Levy [14] defined that these platelets are deformed lips. He also showed experimentally that it is not the micro-machining that forms the lips. In the place, the extrusion is largely responsible in the formation of the lips of the platelets.

2.7.2.1 CUTTING MECHANISM

In the layman language, the mode by which material can be removed from the surface of the bulk material is termed as “cutting”. It is when a hard particle cuts through a softer material, this is known as Cutting wear. Generally, when the impacting particle contacts the target material at positive rake angle along with generating a chip or a cut, the cutting mechanism operates by the material resulting in the generation of new surface. The model of the cutting mechanism is shown in the fig.2.10

There are various models being developed for explaining this mechanism. The surface of the softer metal is ploughed by hard particles of the counterpace according to the abrasive

wear model [15]. It is assumed in this model that an abrasive particle leaves a wear track of the same cross-sectional shape as that of the particle.

It can be shown that without plastic deformation, the wear particles in the form of chips could be generated. The specific energy U (i.e.; the work done to remove a unit volume of material by a cutting mechanism) is equal to the hardness of the material. The equation 2.2 can be written as:

$$K = \frac{3\mu VH}{\mu LS} \approx 3\mu(VU/FS) \quad \dots\dots 2.5$$

Where μ is the coefficient of friction and assuming that by $3\mu = 1$, above equation may be expressed as:

$$K = \frac{VU}{FS}$$

$$= \frac{\text{work done to create abrasive wear particles by cutting}}{\text{work done by the external}}$$

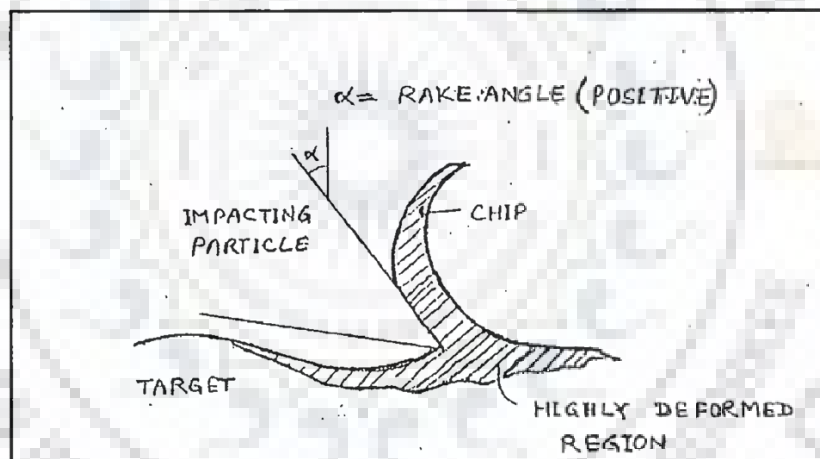


Fig 2.10 Cutting mechanism [8]

It is clarified that K is simply the ratio of the work done to generate wear particles, in the form of cutting chips, to the total external work done. Therefore, the wear coefficient should be equal to or greater than unity when the entire work done is consumed to cut the surface. However, through experimentations, it is determined that maximum coefficients are one or two orders of magnitude less than the unity.

2.7.2.2 PLOUGHING MECHANISM

As a consequence of ploughing, wear particles in elastoplastic solids were also generated. By actions of the wear particles, hard particles entrapped from the environment and by the hard asperities of the counter face, surface can be ploughed. The mechanism involves the displacement and extrusion of the material with no new surface generations, when the particle contacts the target work piece at negative rake angles (figure 2.11). In addition, when considered in terms of the consumed energy to remove the material, the ploughing mechanism is less efficient than the cutting mechanism. Winter et. al. [16] focuses on the role of the rake angle that is important to cutting or ploughing mechanism.

Ploughing can generate chips through the formation of ridges which deform and fracture, due to subsequent asperity interactions.

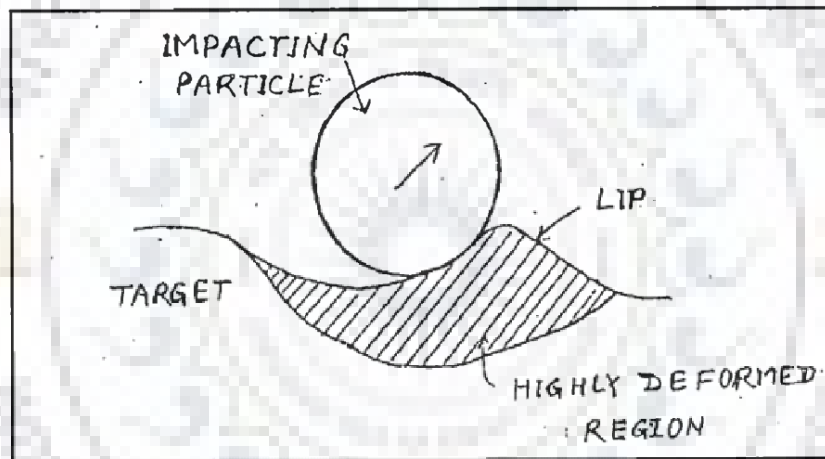


Fig 2.11 Ploughing Mechanism [8]

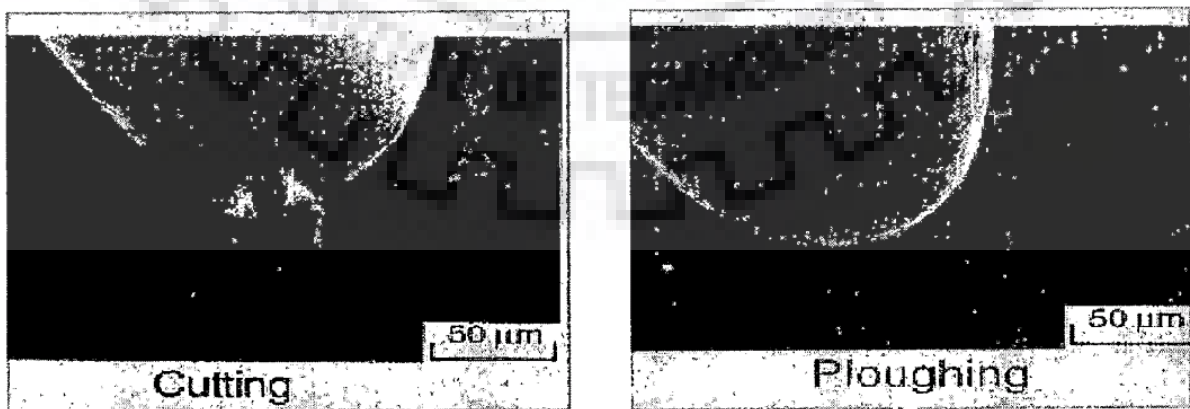


Fig 2.12 SEM microstructure observed of wear process during wear of lubricated brass by steel pin [17]

2.7.2.3 EXTRUSION AND FORGING MECHANISM

In the experiments done by Bellman et. al. [18], the evidence was obtained that extrusion is the initiation of mechanism of platelet erosion. They described that the loss of metal from an eroding surface appears to occur by a combined extrusion forging mechanism. It is indicated from the evidence that the platelets are initially extruded from the shallow craters made by the particle impact. They are forged into a distressed condition when once formed, in which they are vulnerable to be knocked off the surface in one or several pieces.

2.7.2.4 SUBSURFACE DEFORMATION AND CRACKING

Damages are caused in the subsurface of the target by the impact of the silt particles. These have been demonstrated by investigators [8, 19]. With the help of either ploughing or cutting, subsurface may be deformed which can generate chips or wear particles. The transition of wear from abrasion to delamination or sliding wear occurs when subsurface deformation, crack nucleation and propagation process can generate wear particles faster than the rate of chip generation. When the force due to ploughing by wear particles are transmitted from the surface to the bulk, the material at the subsurface undergoes various physical changes.

2.7.3 EROSION BY LIQUID PARTICLE IMPINGEMENT

The Cambridge Team of Bowden and Field [20] of Britain categorized the phenomenon of multifaceted erosion due to the impact of liquid. Very high stresses could arise during the liquid impact, so that even on single impact, various modes of failure such as pitting, scouring or fracture are possible. By repeated impacts, these effects are generally magnified. Moreover, the phenomenon of fatigue often arises. Cavitation erosion arises due to the tensile stress [8] caused by the collapse of bubbles contained in the liquid. Cavitation erosion is alternatively termed as Liquid erosion.

2.8 FACTORS INFLUENCING EROSION

Various factors affect the erosion rates. Some of these factors can be divided into groups.

1. The general factors, such as impact velocity, impact angle, particle shape, particle size, particle density, particle hardness, particle concentration, temperature, medium of flow, load, sliding speed etc. and
2. The metallurgical factors such as hardness, toughness, carbon content, microstructure, temperature, heat treatment and cold work, chemical composition etc.

With the metallurgical treatments, the second type of factors can be controlled. The various effects of these factors that can influence the erosion are explained below:

2.8.1 GENERAL FACTORS

The factors that effects the erosion rates are described below:

2.8.1.1 IMPACT VELOCITY

Since it has a dramatic effect on erosion rate, impact velocity of the abrasive particle (V) is the most important variable. The proportionality has been found between the erosion and the power of the impingement velocity. With the various experiments [8, 21] done on metals and alloys, it has been clearly established that erosion E can be expressed in terms of impact velocity as:

$$E = KV^n \quad \dots\dots 2.7$$

Where K is a constant and n is the velocity exponent. The value of n lies in the range of 2 to 3 in case of erosion of metals and alloys. Hutchings [22] carried out a detailed analysis of most of the erosion data on metals and alloys. In that, it is indicated that foe oblique impact the mean value of the velocity exponent is around 2.4.

On the other hand, Sundaranjan and Shewmon [23] also carried out a similar analysis giving a mean value for n of about 2.55 for normal impact. The reported numerical values of the exponent range from about 1.7 to 2.8 for ductile materials and 1.4 to 5.1 for brittle solids.

2.8.1.2 IMPACT ANGLE

On the type of the material undergoing erosion [24], the influence of the impact angle largely depends. The maximum damage occurs at low impact angle range of 15° to 30° degrees for the ductile materials whereas for the brittle materials, it occurs at normal impact angle ($\Theta = 90^\circ$). The significant erosion occurs even at normal impact angles in the case of ductile materials (fig.2.13). Generally, the erosion rate at 90° is about 20 to 50 pct. Of the maximum erosion rate obtained in the angle range of 15° to 30°. Presently, there is a lack of sufficient data regarding the effects of impact velocity, temperature and particle size on the variation of erosion rate with impact angle.

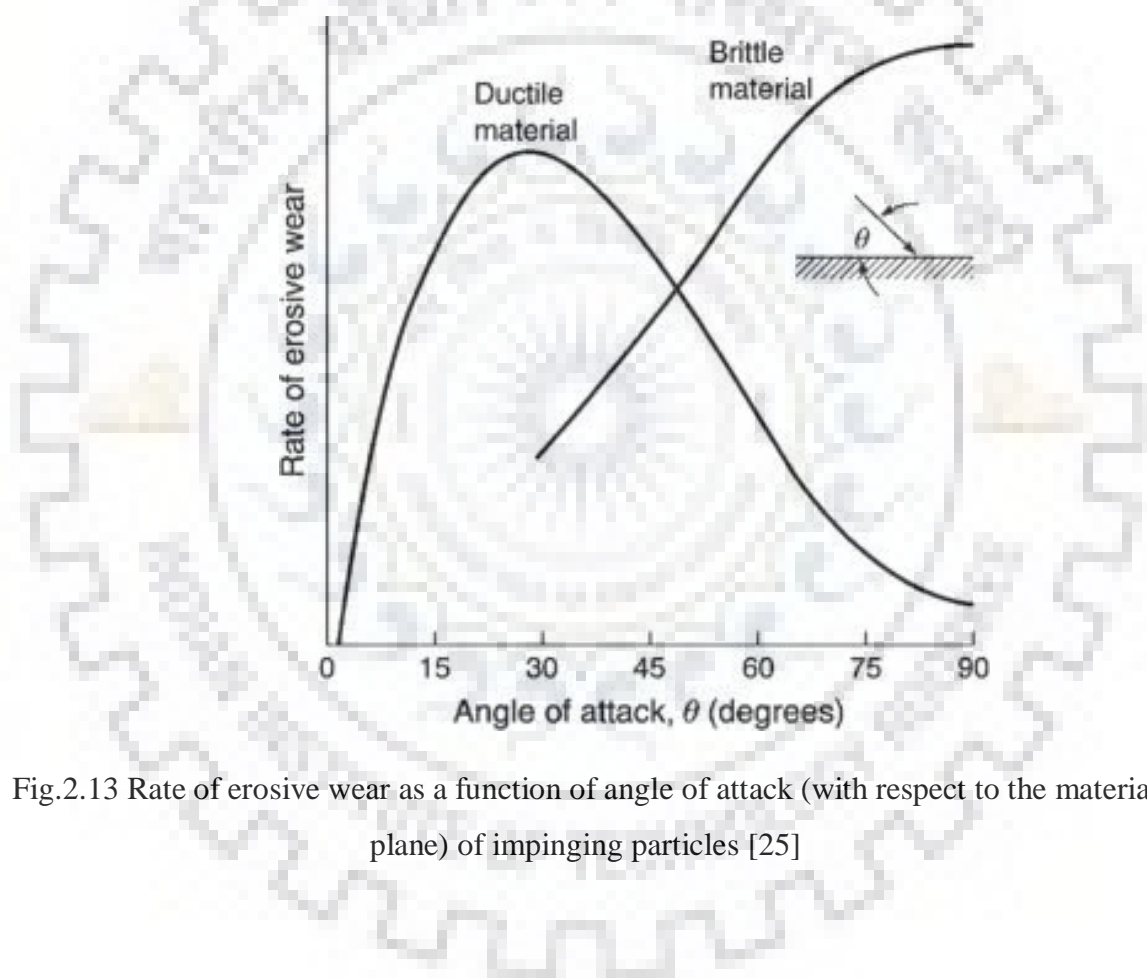


Fig.2.13 Rate of erosive wear as a function of angle of attack (with respect to the material plane) of impinging particles [25]

2.8.1.3 PARTICLE SIZE

There is a rapid increase in erosion with the grit size upto some critical diameter (50 μ -100 μ) and then it increases at a much slower rate or remains constant. This has been attributed to abrasive deterioration and embedding of abrasive with the decrease in abrasive size. Wear is no longer caused by the abrasive wear mechanism when the particle size is less than 1 μ but

begins to approach the delamination wear. Thus, this is the reason that why it is not clarified for the very small particles being so effective in causing the erosion. It has been found that normally brittle materials will erode in a typically ductile fashion with sufficiently small particles.

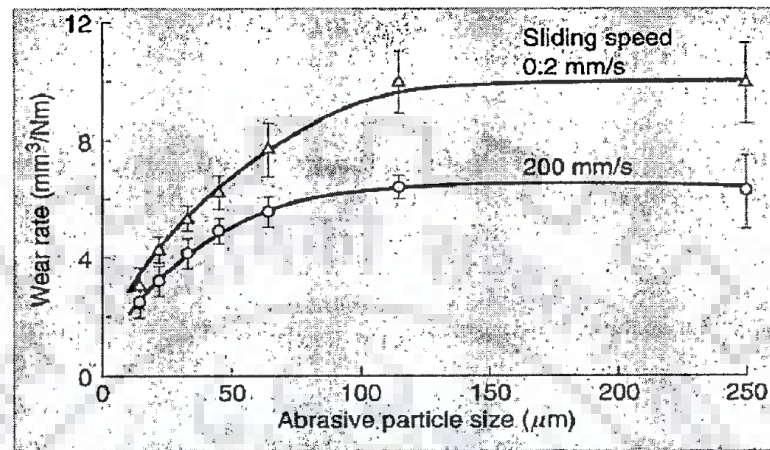


Fig.2.14 Wear rate of copper, subjected to two body abrasion by SiC abrasive, as a function of abrasive particle size at two different sliding velocities. [26]

2.8.1.4 PARTICLE SHAPE

In general, it is found that instead of the spherical particles [27], angular particles are more effective in causing the erosion. In addition, when the angularity of the particle is more, the impact angle at which the maximum erosion occurs is generally low.

2.8.1.5 PARTICLE CONCENTRATION

Young [28] had studied the effect of particle concentration. They concluded that the erosion rate should be independent of concentrations of particle so long as each impact of particle event is equally effective. The erosion rate should decrease at high concentration where particle interference effects occur. With the increasing total mass impact, the erosion also increases. Therefore, the relationship in most of the cases is linear. However, the efficiency of the process of erosion is quite low. Erosion rate per unit mass increases with the decrease in particle flux.

2.8.1.6 PARTICLE DENSITY

Generally, when the density of the imparting particle is high, the rate of erosion is greater. Compared to the soft particles, the hard particles like quartz is more erosive.

2.8.1.7 SPEED OF SLIDING SURFACE

With the change in speed, erosion rate could change considerably. But however, between speed and erosion rate, there is no general relationship. Depending on the effects of the temperature of the surfaces, an increase in speed can lead to an increase or a decrease in erosion.

2.8.1.8 TEMPERATURE

Erosion tends to be fully dependent on the temperature [29]. In general, as we know, the erosion rates increase with an increase in the temperature because with the increasing temperature, the material involved becomes softer.

Several investigators had examined the effects of temperature on the erosion rate but the results are not mutually consistent. It has been observed by Gat et. al. [30] that in the case of metals, below about 0.3 T_m (Target melting point) erosion resistance increases with the increase in temperature. While above 0.5 T_m, the erosion resistance decreases with the increasing temperature.

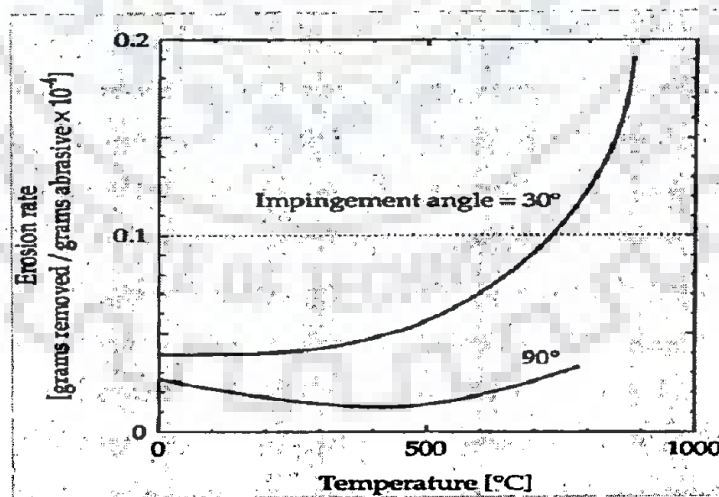


Fig. 2.15 Effect of temperature on erosion of stainless steel

In the single-phase metals, the erosion increases with the melting temperature of the work piece. It has been found by Ives [32] that a significant increase in the erosion rate on going from 25°C to 975°C in the case of 310 S.S. targets impacted at 90°. In the case of another study done by Finnie on the pure aluminium revealed that the effects of temperature were quite complex. Erosion rate decreases with increasing temperature up to 0.6 tm at low impact angles and at high temperature erosion rate increases. At higher angle of impact, on the other hand, it is observed that the erosion rate attained a minimum at around 0.4 Tm itself. There is one exception to the general temperature effect that the erosion of very hard material such as nickel tends to increase with increasing temperature especially as the nominal impingement angle increases. Thus, there is lack of sufficient data being present in the literature regarding the effect of target temperature on the erosion rate.

2.8.2 METALLURGICAL FACTORS

Mechanical properties and metallographic structure are the main metallurgical factors affecting erosion.

2.8.2.1 MECHANICAL PROPERTIES

While selecting the metals for minimum erosion, metal hardness and toughness are among the most important characteristics that must be considered. In specific applications of various materials, hardness appears to be a dominant factor governing the erosion resistance. In general, as the hardness increases, wear resistance also increases and it decreases as the toughness increases. This is an important relationship in application that requires both wear resistance and impact resistance. Finnie et. al. [33] has correlated the erosion resistance of ductile metal with their hardness. As a result, hardness serves as a good indicator of erosion resistance only in the case of pure metals. In the figure 2.16, the correlation between wear resistance and hardness of various class of material has been shown. From the diagram portrayed below, on can observe that the erosion rate of the pure metals is inversely related to its hardness.

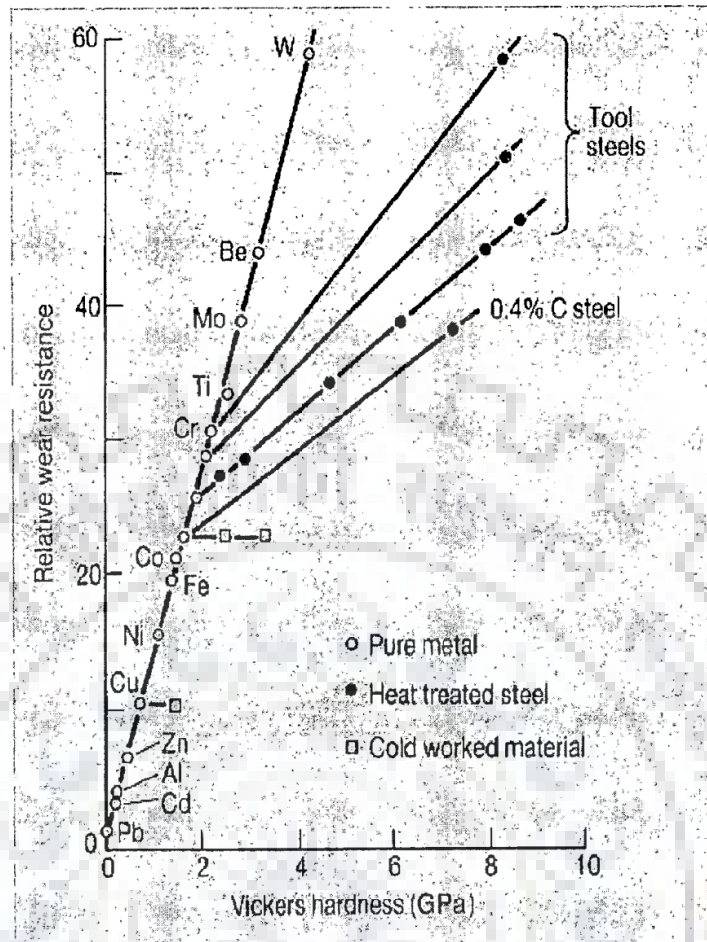


Fig. 2.16 Relative wear resistance of pure and heat treated and cold worked steel as a function of hardness in two-body abrasion [26].

In the fig.2.17; the linear plot shown for the pure metals as the steep line and another straight line brittle ceramics reasonably. However, for in the case of two phase metals, it is insensitive to both heat treatment and cold working. Therefore, the hardness of two phase metals does not correlate with their erosion resistance. Finnie [33] provides a fine example through his experimental result. It is indicated from these results that increasing the hardness of tool steel and 1045 steel by a factor of five through heat treatment has no effect on their erosion. Moreover, the erosion resistance of forty different metals and alloys had been examined by Soderberg [34] in his study. From their results, it is indicated that solid solution strengthening, quench hardening, and precipitation hardening etc. does not results in any improvement with regard to erosion resistance.

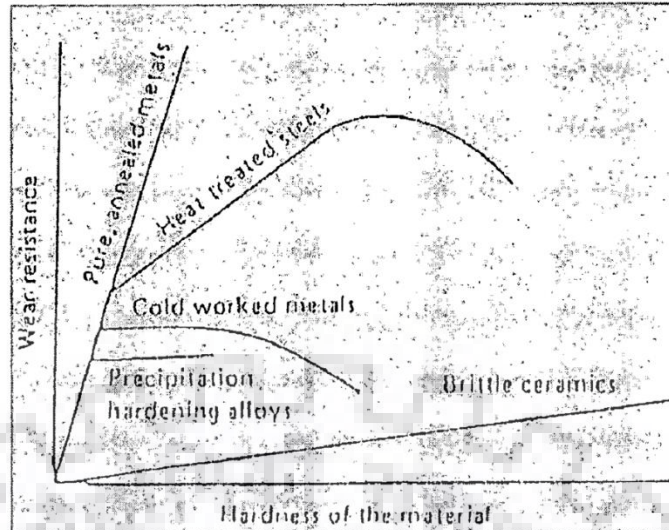


Fig. 2.17 Schematic representation of the correlation between wear resistance and hardness of target materials in various microstructural conditions [8].

A correlation between wear resistance and toughness is also important. With the microstructure, toughness is generally affected which in turn also affects the erosion behaviour. The rate of erosion is affected by the toughness of the metals which is closely related to the crack propagation. The material will have more resistance to erosion and also the crack propagation if the material is tough. Similarly, the material will have resistance to indentation or scratching or lower will be the erosion rate if the material is sufficiently hard. In the two-phase metals, cracks are more prevalent to occur. There can be non-occurrence of cracks and the erosion rate shall be low in the high purity single phase metals.

A number of papers have been appeared in the literature [33, 35] on the effect of mechanical properties on the solid particles. An attempt was made by Foley et. al [36] in his study to characterize the erosion behaviour as it relates to the mechanical properties obtainable in alloy steel by conventional heat treatments. Ductility was an important parameter. This is indicated from the erosion test on hot-rolled 1020 and cold rolled 1020 steels and annealed 304 and wrought 304 stainless steels. It was found by them that the erosion resistance increases with the increasing ductility and that the hardness, fracture, strength, toughness and the impact strength had little effect on the erosion.

The erosion behaviour was studied by Naim et. al. [37]. He studied the erosion behaviour of 18 Ni miraging steel and concluded that the erosion rate depends on the hardness and ductility both. When the ductility remains constant, the erosion rate varies directly with the hardness and it varies inversely with square of pct. area reduction when hardness remains constant.

Depending on the hardness, the erosion resistance of material increases or decreases to different extents or remains unchanged [33, 38]. It was concluded by Lou et. al. [39] in his investigation on the seven most utilised materials that the erosion resistance increases to a maximum and then, decreases with decreasing material hardness. In regard to the strain, the wear resistance is related to the fracture toughness that occurs during asperity interactions compared with critical strain at which crack growth is initiated.

Goretta et. al. [40] has demonstrated in his study that the solid particle erosion behaviour of AISI 4140 steel heat treated to have Vickers hardness of 288-650 VHN. They concluded that the erosion rate was nearly independent of hardness for $VHN \leq 365$ but increase with hardness of $VHN \geq 365$. Also, with the increased ductility, the improved erosion of the softer alloys was attributed.

A research was carried out by Schumacher [41] on the wear and erosion of various steels and found that the erosion behaviour is inversely proportional to the hardness. For example, when tested the wear resistance properties were not exhibited by AISI 4230 even on increasing its hardness. On the other hand, with the increasing hardness, the wear and erosion resistance of 17-4 PH steel does not always improve. Schumacher stated that concept “the higher the initial hardness, the better the erosion resistance” is almost untrue for high strain hardening alloys.

As learnt from the literature so far, the variation of erosion with mechanical properties does not seem to follow any universal pattern. In general, with the increase in hardness or the tensile strength, erosion tends to increase and it decreases with the improvement in ductility [42].

2.8.2.2 MICROSTRUCTURE

Metallurgically, microstructure and the hardness are commonly interrelated and both have importance as factors in resistance to erosion. The increment in the carbon content of carbon steel results in microstructure attention that increases as quenched hardness and

decreases the ductility and toughness. On the amount of the carbon content of steel [43] and the amount of martensite, the maximum hardness shall depend.

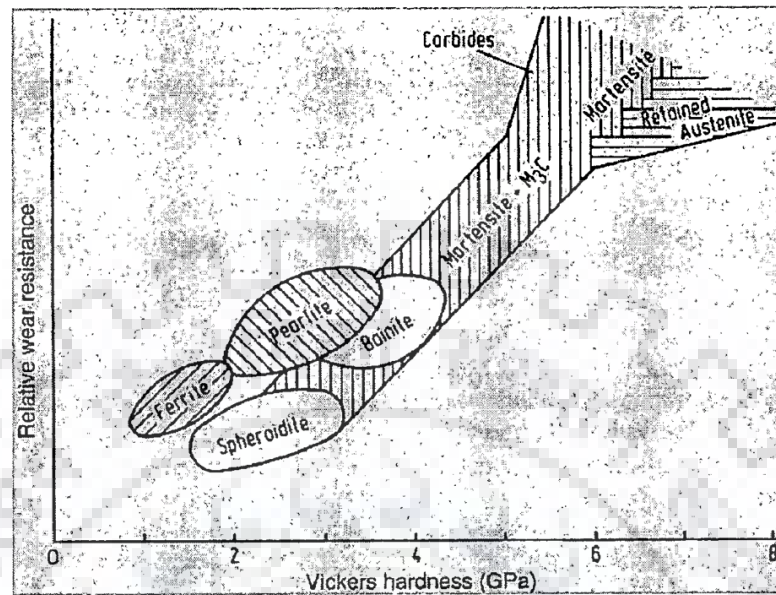


Fig.2.18 Relative wear resistance as a function of hardness of different microstructures of steel [44].

It is indicated from Standard hardness measurements that steel is largely transformed, although it may retain some austenite. The exposure by tempering can help complete the transformation to martensite and improves erosion resistance. Through the formation of various simple and complex carbides, carbon content affects the hardness and erosion resistance. There are several factors on which the erosion properties depend upon such as type, amount, size, shape and distribution of carbides present, as well as the properties of the matrix. Correlation of erosion rates with the microstructural constituents is possible despite of any complexity.

Various authors have studied the effects of microstructure on erosion [8, 33]. The erosion of AISI 1045 steel in the annealed, hot rolled and water quenched (tempered and untampered) conditions impacted by a stream of SiC particles have been studied by Finnie. Resulting from the above heat treatments, they observed a slight decrease in erosion resistance with a fourfold increase in hardness. They also found that the erosion resistance of type 01 tool steel was the highest in the condition, lower on oil and still lowering on tempering.

Salik et.al. [38] Has performed the erosion studies. He tested on the AISI 1045 steel in annealed, spheroidized, normalised, austempered, water quenched and tempered conditions. They concluded that the metallurgical changes affected the erosion resistance more

significantly than the changes in hardness. Sargent et. al. [45] eroded AISI 1050 and 1080 steels under normalised and water quenched conditions with Aluminium oxide and coal ash particles. Except in the 30°-50° angle impingement range, in which case 1080 steel in water quenched condition was slightly less erosion resistant than in normalised condition, they found the erosion resistance of these steels to Aluminium oxide was about same. AISI 1080 steel was more erosion resistant to ash particle than 1050 steel in the water quenched condition.

Using SiC particles, Levy [43] studied the erosion of AISI 1020 and 1075 steels. The spheroidized microstructure was found to be more resistant than the pearlitic structure in the case of 1075 steel. A higher erosion resistance was provided by the pearlitic microstructure with coarse pearlite than the microstructure with fine pearlite. It has been implied from both of these observations that the erosion resistance decreases with the increasing hardness. It is found that the erosion resistance of 1020 steel is dependent ambiguously upon the distribution of hard brittle Θ phase and soft ductile α phase.

In the systematic study of Aptekar et.al. [46] of white cast irons with different volume fractions of carbide showed that the erosion behaviour of these materials was sensitive to the properties of the erodent. Using alumina as the erodent (higher than the carbides in the iron), higher carbide volume fractions were detrimental to the erosion resistance, whereas quartz erodent (softer than carbides) showed higher carbide volume fractions to be beneficial. However, the white iron eroded faster than annealed 1020 steel, even for the quartz abrasive. It indicates the approach of using second large phase carbides to be ineffective in erosion despite its utility in the abrasion. In the work done by Andrew Ninham [47] on the erosion, he concluded that the carbide particles which confer abrasion resistance to alloys were found to be disrupt plastic flow around the indentation, causing void formation and fracture. Thus, in context of erosion performance, the carbides were deleterious.

Various studies had been done on the erosion behaviour [35, 37, 42 and 43] in which they have provided some basic understanding with respect to the popular microstructures as found in steels and aluminium alloys. For example, for the erosion resistance, primary martensite is the worst as it is the hardest microstructure in steel. For the improvement of erosion resistance, tempering of martensite should be done. The loss of strength and spheroidization are the results of excessive tempering which is excellent for the erosion [39]. Initially, precipitation results in the loss of erosion resistance in case of aluminium alloys and

precipitation hardening maraging steel [42]. However, the over ageing contributes to the improvement in the erosion resistance.



FORMULATION OF PROBLEM

This study has been done to improve the life and erosion resistance of underwater parts in the hydroelectric and various projects which are made up of 13/4 martensitic stainless steel. For improving the life of the material, the possible method would involve the attainment of uniform microstructure without any heterogeneity through a simple heat treatment procedure which involves the temperature higher than that of AC3 temperature. In this review, various investigations have been carried out indicating that the erosion resistance of the material could be improved by altering the microstructure. During the erosive conditions, uniformly tempered martensite gives the better performance. Therefore, it was decided that under investigation, the material should be given the suitable heat treatments to attain the tempered martensitic microstructure.

For this purpose, the austenitizing treatments had been given to the steel in as-cast condition at temperature of 950°C, 1000°C and 1050°C with the holding time as 2hrs, 3hrs and 4 hrs for each temperature. The similar method would be followed in Oil quenching. The tempering treatment would be given to the oil quenched samples which involves heating the specimens at 600°C for 1 hour. A detailed study had been conducted on the as cast material with a view to find out the various changes in the mechanical properties, microstructures and erosion on the heat treatments. Toughness, hardness, UTS and ductility i.e. pct. elongation was some of the mechanical properties determined through this. The studies on the erosion have been conducted by determining weight loss by impact of sand particles on the surface of the specimens in a laboratory slurry testing machine.

Erosion testing machine has been used for the test of erosion. In this the samples have been mounted in between the plates held at different angles, tangent to the direction of the slurry i.e. 0°, 30°, 60° and 90° and the samples rotate in the slurry. The concentration of the sand particles in the slurry is 15%. Also, the erosion rate is determined according to the weight per unit surface area loss.

EXPERIMENTAL PROCEDURE AND TECHNIQUES

4.1 MATERIAL USED

In this investigation, the material being used is 13/4 martensitic stainless-steel conforming to the specification ASTM A743 CA-6 NM. The chemical composition of the martensitic stainless steel in weight % is as follows:

Composition pct. Max.								
C	Mn	Si	Cr	Ni	P	S	Mo	Fe
0.06	1.00	1.00	11.5-14.0	3.5-4.5	0.04	0.04	0.4-1.0	balance

4.2 HEAT TREATMENT

The heat treatments are given using the furnace silicon carbide muffle furnace. Standard heat treatments were given to the cast CA-6NM steel (13 Cr-4Ni martensitic stainless cast steel).

For studying the effects of heat treatment, 6 samples of size 100mm x 25mm x 25mm were heat-treated in 2 sets each having 3 samples at a time, at the given temperature and soaking time. The martensitic steel may cause warpage and cracking due to low thermal conductivity and high stresses during rapid heating. Preheating was carried out from 550°C to 1000°C @ 100°C/hr for avoiding these problems.

Austenitization was carried out at 950°C, and 1000°C which was considered high enough to allow dissolution of carbides without excessive grain coarsening. For each austenitizing temperatures, the soaking time for every set was 2 hrs, 3hrs and 4hrs.

Austenitizing is followed by oil quenching and finally tempering it for 1 hour at 600°C for each sample. The table 4.1 shows the various heat treatments performed on 13/4 martensitic stainless steel

Table 4.1 Heat treatment at various temperatures

S.no.	Treatment/Conditions	Tempering	Designation
1	As received	-	AR
2	Austenitized at 950°C for 2 hrs. followed by oil quenching.	Tempered at 600°C for 1 hour	950°C-2hrs-OQ-600°C-1hr
3	Austenitized at 950°C for 3 hrs. followed by oil quenching.	Tempered at 600°C for 1 hour	950°C-3hrs-OQ-600°C-1hr
4	Austenitized at 950°C for 4 hrs. followed by oil quenching.	Tempered at 600°C for 1 hour	950°C-4hrs-OQ-600°C-1hr
5	Austenitized at 1000°C for 2 hrs. followed by oil quenching.	Tempered at 600°C for 1 hour	1000°C-2hrs-OQ-600°C-1hr
6	Austenitized at 1000°C for 3 hrs. followed by oil quenching.	Tempered at 600°C for 1 hour	1000°C-3hrs-OQ-600°C-1hr
7	Austenitized at 1000°C for 4 hrs. followed by oil quenching	Tempered at 600°C for 1 hour	1000°C-4hrs-OQ-600°C-1hr

4.3 TENSILE TEST

The tensile test was performed on a “MONSANTO” type W. TENSOMETER using a cross head speed of 1mm per minute. According to the formula mentioned below, the % elongation is calculated.

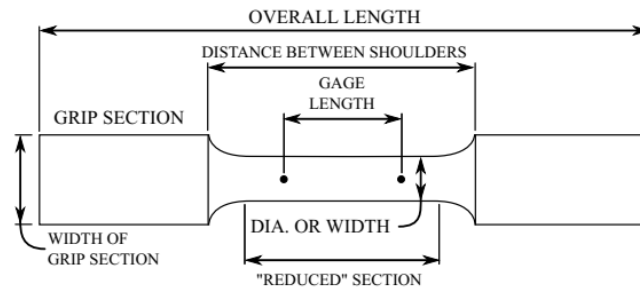


Fig 4.1 Tensile test specimen

Fracture load, proof stress, and % elongation, ultimate tensile strength was found out from the stress-strain curves values of yield strength. With the help of digital vernier calliper, the total gauge length was measured. With the help of the given formula, the theoretical value of % elongation was calculated. The formula is:

$$\% \text{ Elongation} = [(L_F - L_O) / L_O] \times 100 \quad \dots 4.1$$

Where,

L_F = final gauge length

L_O = initial gauge length

4.4 HARDNESS TEST

Diamond was used in the Vickers hardness test, with the shape of square-based pyramid with an angle of 136° between opposite faces as an indenter (22° between the indenter face and surface). This is based on the principle that regardless of the load, the impressions made by this indenter are geometrically similar. Therefore, accordingly, to a flat surface, loads of various magnitudes were applied depending on the hardness of the material to be measured. The tests of the hardness were performed on a BHN cum VHN hardness Testing machine.

By measuring the two diagonals of the square indent, the size of the indent was determined. The calculation of the mean of the two diagonals was done and determination of the corresponding hardness could be done using the following formula:

$$\text{HVN} = (1.854 P / d^2) \text{ Kg/mm}^2 \quad \dots 4.2$$

Where,

P = Load (10 Kg)

d = Arithmetic mean of two diagonals, d_1 and d_2 in mm

4.5 IMPACT TEST

The impact test is done to measure the impact energy, or the energy absorbed prior to fracture. Charpy test and Izod test are the most common methods of measuring the impact energy. For measuring the toughness of the material and the yield strength, the quantitative results of the impact test can be used. Not only the quantitative results, the qualitative results of the impact tests are also of great usage. Hence for determining the ductility of the material, the qualitative results of the impact test can be used. The fracture is brittle if the material breaks on a flat plane. Also, the fracture is ductile if the material breaks with jagged edges or shear lips. Usually, in just one way or the other, a material does not break. Thus, only an estimate of the percentage of the ductile and brittle fracture could be estimated from comparing the jagged to flat surface areas of the fracture.

In the Charpy test, the specimens normally measure 55 x 10 x 10mm and have a notch machined across one of the larger faces. A V-shaped notch was made and tested being 2mm deep, with angle of 45° and 0.25mm radius along the base of the specimen.

4.6 MICROSTRUCTURE

A study has been conducted on the microstructure of the steel which was received as along with the specimens on which heat treatment were done. These specimens were having minimum weight loss at different temperatures. The following steps are to be followed in order to prepare the sample.

1. On the grinding machine, the specimens were grounded.
2. After this, the polishing would be done on the belt grinder.
3. Then the polishing would be done with the help of the series of emery papers. i.e. 1/0, 2/0, 3/0 and 4/0.
4. Thereafter taking the help of machine of cloth polishing, the sample was polished. For this, ferric oxide which is red in colour is used along with water.
5. The ferric chloride in methanol reagent is used as the etching reagent. The ferric chloride in methanol reagent is prepared by mixing 5 grams of ferric chloride with 100 ml of methanol. Thereby the etching shall be done by using this reagent.
6. The optical microscope shall be used for examining the etched sample being properly polished.

4.7 FRACTOGRAPHIC STUDIES

With the help of scanning electron microscope, specimens were examined pertaining to the ones fractured in the Charpy tests and in the tensile test for gaining the insight into the fracture behaviour. Therefore, for this, a small section of specimen was cut out from the fractured specimen very carefully in order to curtail the fractured specimen from any damage.

The wheel grinding is used for flattening the surface which is to be examined. This surface is being opposite to the fractured surface. Thereafter, a silver paste is used for gluing the sample. This sample is carried carefully and is placed and glued properly in the specimen holder of SEM. Scanning is to be done of the fractured surface thoroughly. The interest points were photographed diligently. Chapter 5 demonstrates the results of the Charpy test and the tensile tests. A detailed presentation and discussion is done in the aforesaid chapter.

4.8 EROSION TEST

From the era since the middle of 20th century, the studies of the erosion explanatory with the experiments were already been started. For the academic and research centres across the world, several methods have been evolved for measuring the erosion. These methods are currently being in use too. The dynamic operating conditions are responsible for the evolvement and usage of these methods.

Summing up the facts mentioned, between the mixture and the specimen, the relative motion that too between them. On the basis of which, there are three types of the typical and general erosion testing rigs. The names of these rigs are as follows:

- (a) One of them are rotating disc of arm type.
- (b) The Another one is jet or nozzle type.
- (c) Also, the centrifugal accelerator type.

4.8.1 DESCRIPTION OF EROSION TESTING MACHINE

The diagram displayed in the figure 4.2 is the machine for testing the erosion. This machine is recognized as Slurry pot erosion tester. A container is being comprised by the

machine. The slurry is poured onto this container. The 15% wt. that is around 12.5 vol% of concentration of slurry fills up the container. In order to minimize the rotational flow of the bath of the slurry, the container of the slurry comprises of the 4 plates of baffle from the inside. The electric motor is used in order to drive spindle at 2515 rpm (263 rad/s) along with taking the help of belt which is V-shaped. This belt imitates an immediate cylinder velocity at the rate of 14.8m/s. A central shaft is embraced by the spindle which was involved in the sample holder. Through the gear level mechanism, at any position, such as being affixed vertically, the central shaft is easily modifiable and can be adjusted accordingly.

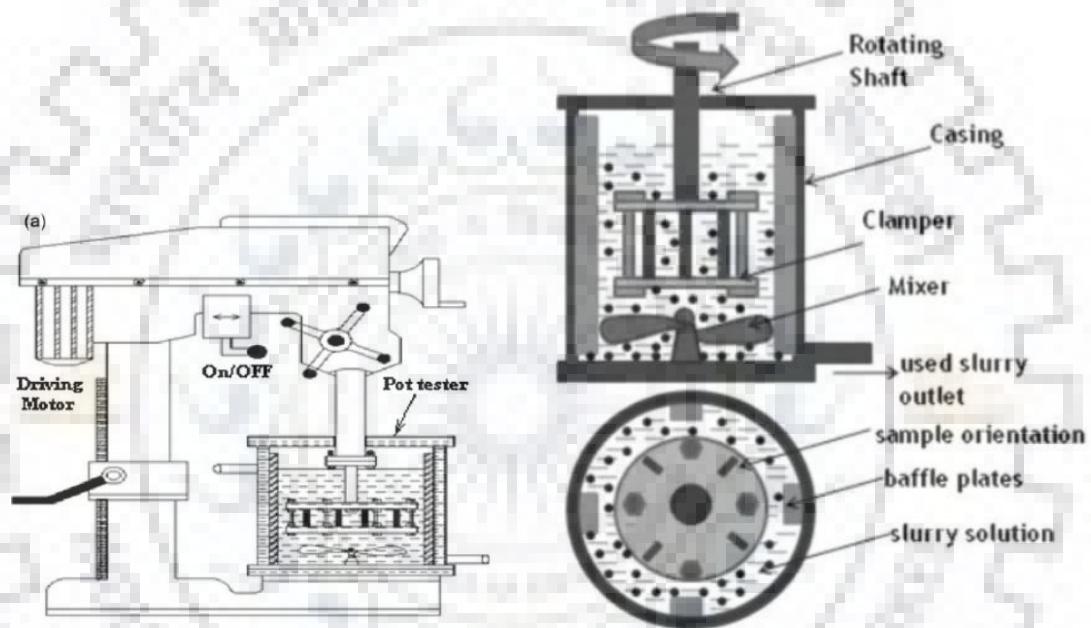


Fig. 4.2 Schematic of slurry pot erosion tester.

There are two disc plates being comprised by the sample holder. These plates were placed close to each other together along with the arrangement of four bolt and nut. These are used to clamp the sample. The slots at the angles that is 0° or 30° or 60° and 90° were being cut at that particular angles being tangent with respect to the slurry. The slots were cut on both top and the bottom part of disc plates from inner side to accommodate the sample. This can be better illustrated through the diagram shown in the fig.4.2.

4.8.2 TESTING METHOD

The slurry erosion test does not use the mechanised form. Hence, the activities under the slurry erosion test are generally in manual form. The experiments are performed and their observations are recorded manually. The procedure of test is given below in chronological order.

1. Firstly, for the erosion test, the belt grinder is used for preparing the specimen. The polishing is done on the emery papers 1/0 and 2/0.



Fig 4.3 Specimen for erosion test

2. Thereafter the specimen is cleansed by using acetone.
3. The specimen is dried out properly.
4. At this time, before moving further, jot down the specifications of the specimen such as the original length, height, breadth or the weight it properly.
5. The preparation of slurry requires the addition of seven parts of water in the one part of sieved sand. In brief, the ration will be of 7:1 of water and sand respectively in a container.
6. At the various angles of impingement, lock up the specimens in the sample holder.
7. Now the sample holder needs to be attached to the spindle. It has to be immersed in the slurry and thereafter the spindle shall be locked completely.
8. Now it's time to start the machine. Jot down the operating time properly.
9. In each hour interval, the specimen needs to be removed from the spindle and from the sample holder.
10. Repeat the second and third steps again. That is, the cleaning of specimen and making it dry for the further procedure on them.
11. Don't forget to note down weight of the specimen after the erosion.

The particles of the sand been sieved on the Tyler testing in a machine. This machine is named as Ro-tap machine and is used for obtaining the ranges of size of interest during the experiment.

The slurry mixture was prepared by using the sample of sieved sand particles. This mixture is made for the experimental run or operation.

An analysis named “Sieve analysis” was being carried out in order to determine the size of the particle. Half kg of sand was used for the sieve analysis. The arrangement of sieves is in the type of a column settled one over the other. At the top of the layer, the coarsest meshing is arranged. Also, in the bottom the finest one is arranged with the decreasing size of the meshing. In the first sieve, sand was kept having the coarsest meshing among all. The sieves were well shaken and therefore it settles down. It generally settles in the sieves which are of different meshing. In each sieve, the sand being restored and retained is weighed properly. Thereafter, the particle size is determined, calculated and noted down.

4.8.3 TEST VARIABLE

In this section, on the pot of slurry erosion tester, test variables for the measurements are reported.

4.8.3.1 TARGET MATERIAL

The targets being used in the present work are of Ten different types. They are specimens of 13/4Cr-4Ni martensitic stainless steel material. Charpy impact test and the Vickers hardness test are used for measuring the toughness along with the hardness of the specimen. The materials being used are listed in the table 5.2.1 and 5.3.3.1. The table also illustrates the hardness and the toughness of the material. It has been noticed that the material’s hardness varies between 386 to 269 VHN. This corresponds to the condition being heat treated. The toughness of the material is also a verifiable factor which varies between 40.22 to 118 J which similarly corresponds as heat treated and received respectively.

Table 4.2 Target material properties

SPECIMEN SPECIFICATIONS	CHARPY	HARDNESS TEST
	IMPECT TEST	
	TOUGHNESS	VHN
	(Kg-M)	P = 10 Kg
As received	4.3	380
950°C-2 hr-OQ-600°C-1 hr	6.0	279
950°C-3 hr-OQ-600°C-1 hr	6.9	230
950°C-4 hr-OQ-600°C-1 hr	8.9	236
1000°C-2 hr-OQ-600°C-1 hr	6.4	314
1000°C-3 hr-OQ-600°C-1 hr	6.2	284
1000°C-4 hr-OQ-600°C-1 hr	5.9	324

4.8.3.2 RANGE OF PARAMETERS

The table 4.3 demonstrates the range of parameters. These parameters are as according to the present investigation. The experiments were carried out at initial level for various combinations of target and impingement angles. It was established to represent the outcome of properties on the material being mechanical in nature on the wear. The particle size ranging was chosen for performing the experiments at the velocity of 14.8 m/s and being concentrated at 16.66 wt%

Table 4.3 Experiment parameters

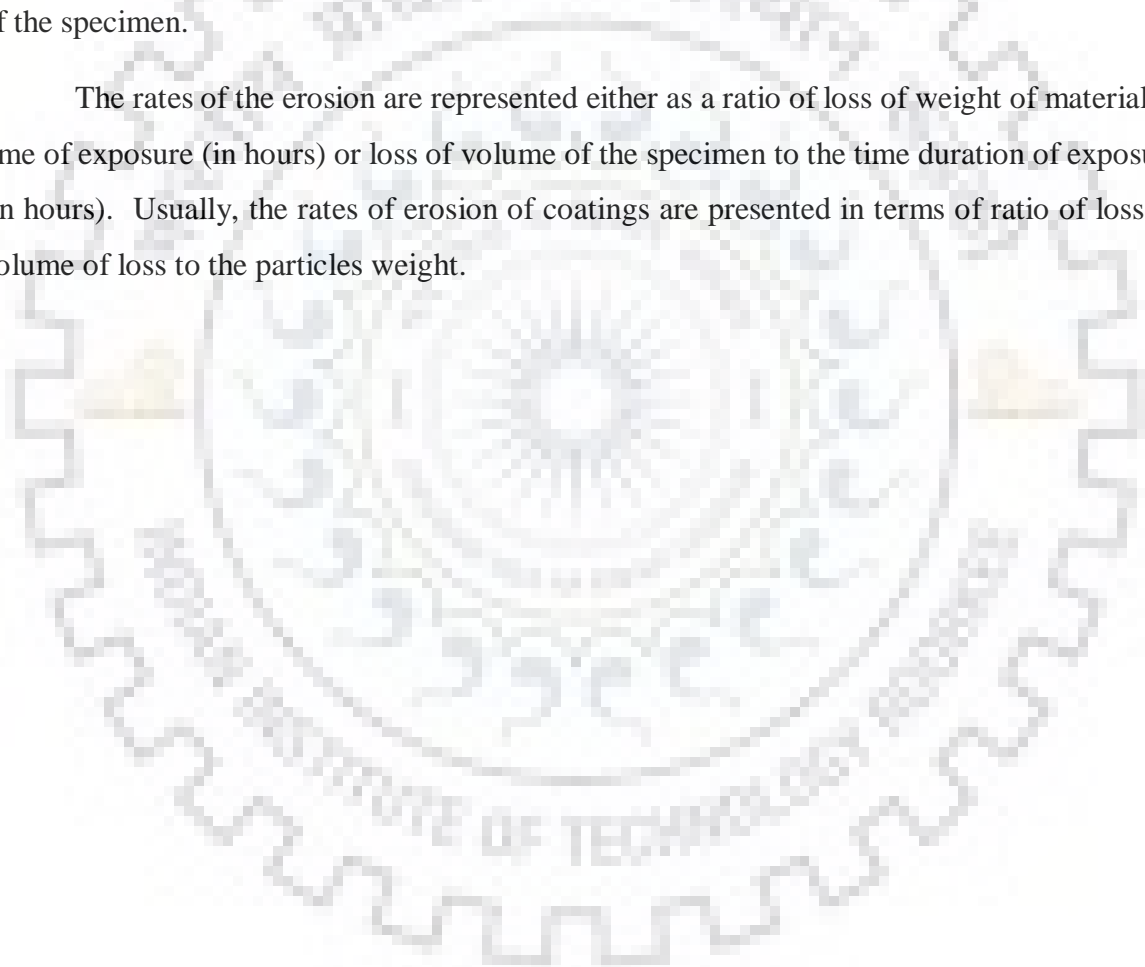
Variables	Operating conditions
Erodent material (1100 HVN)	Quartz
Mean particle size (μm)	48
Solid concentration	15.00
Velocity (m/s)	14.8
Temperature ($^{\circ}\text{C}$)	Room temperature

4.8.3.3 TEST RESULTS

The chapter 5 illustrates the test results from the experiments done on erosion. It took 16 hours to conduct the experiment. In every 4 hour, weight loss is measured for each of the specimen. Moreover, cumulative weight loss is also calculated after every 4 hour while performing the experiment.

In order to ease the comparison and analysis of the data, calculation of weight loss shall act as a key for such purpose. For the calculation of weight loss, the surface area is calculated properly and it shall be divided to the weight loss of the specimen. This gives the results of weight loss in terms of gm/mm^2 . This made the erosion loss being independent from the size of the specimen.

The rates of the erosion are represented either as a ratio of loss of weight of material to time of exposure (in hours) or loss of volume of the specimen to the time duration of exposure (in hours). Usually, the rates of erosion of coatings are presented in terms of ratio of loss of volume of loss to the particles weight.



RESULTS AND DISCUSSION

The chapter 4 comprises of the results of the experiments. The microstructure is analysed and discussed properly in relation to the effects of heat treatments on the mechanical properties of them. There are various studies that has been utilised in order to understand the austenitizing temperature and the tempering duration on the numerous properties of 13/4 martensitic stainless steel. These studies are named as SEM studies or the optical studies. The mechanism of erosion and the subsequent and the subsequent effect of studies on the martensitic stainless steel are also discussed.

5.1 HEAT TREATMENT TEST RESULTS

Table 5.1 and the figures 5.1 and 5.5 presents the effects of heat treatments on microstructure and mechanical properties.

5.1.1 EFFECT OF HEAT TREATMENT ON MICROSTRUCTURE

Under the investigation, the optical microscopic studies were conducted on the steel in as received or the conditions that has been heat treated. Retained austenite, precipitation of carbides and the balance of martensite are the variables that are comprised by the principle microstructure.

In the figure 5.2 (a) pertaining to as received microstructure. It shows the existence of packets made of finely untampered lath needles of martensitic steel. Along with these packets, the presence of particles of coarse carbide (black dots) being located generally prior to austenite boundaries of grain are demonstrated by this structure. The micro analysis of these electron prone carbides is not conducted. However, the literature portraits that the carbide precipitates in the 13/4 martensitic stainless-steel corresponding to the mixture or composition M_3C , M_7C_3 and $M_{23}C_6$

In the figure 5.1(b)-(g), the microstructures are reported for several austenitizing temperatures and for the tempering conditions. Tempering of martensitic needles are caused by the heat treatments. These symptoms are revealed by the microscopic studies. During the autenitizing process, the changes that may occur are as follows:

- (i) When the autenitizing temperature increases, the austenite grain size also increases.
- (ii) At the time of soaking, the dissolution of carbides and the alloying elements also occurs.

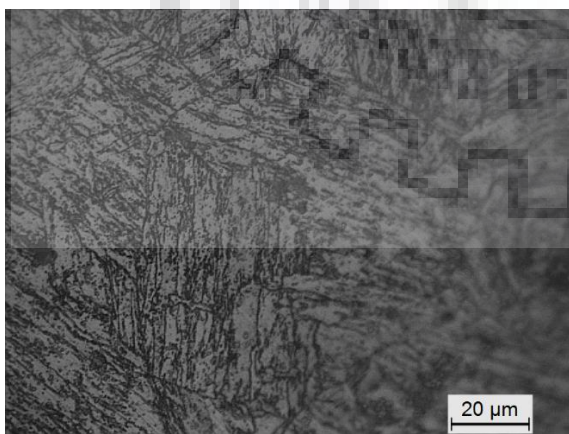
From the micrographs in the figure 5.1 (b)-(g), it is clearly apparent that the alloy carbides are getting dissolved during the heat treatment as compared to the received structure. With the increasing amount of dissolving carbides, the amount of retained austenite also increases. Moreover, the size of lath martensite needle also increases with the increasing austenitizing temperature. This had been revealed by the microscopic studies.



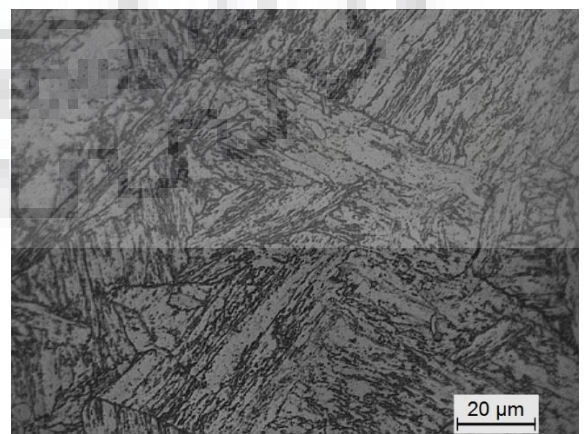
a) As received 13/4 MSS at 50X



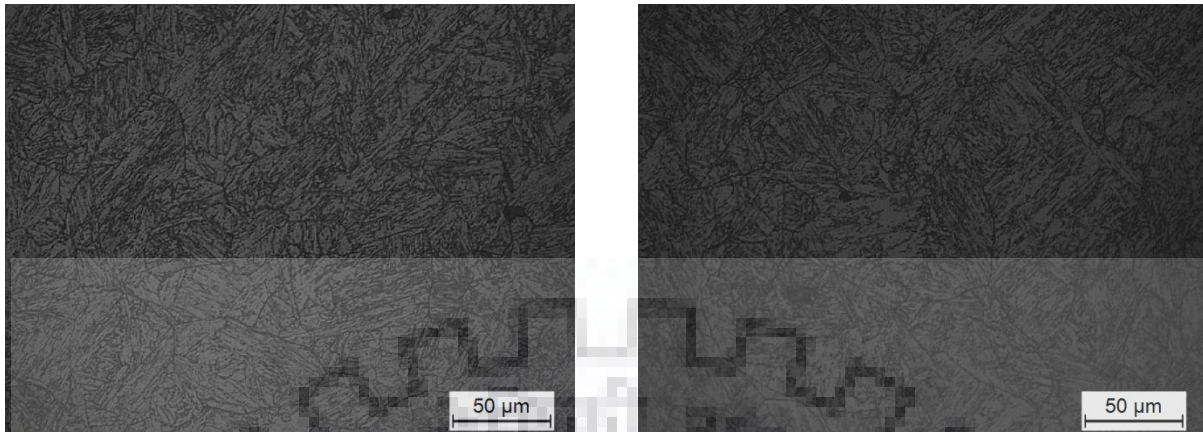
b) 950°C-2 hr-OQ-600°C-1 hr MSS at 100X



c) 950°C-3 hr-OQ-600°C-1 hr MSS at 100X

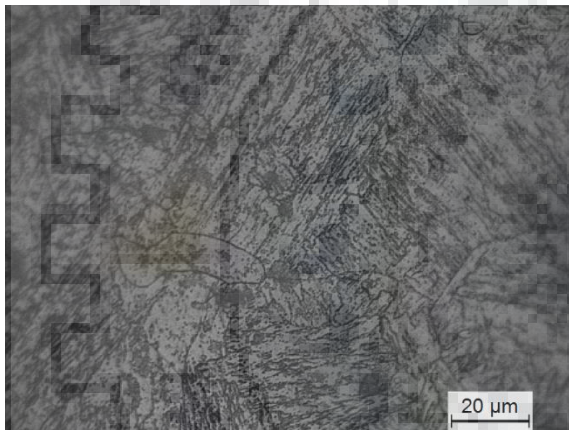


d) 950°C-4 hr-OQ-600°C-1 hr MSS at 100X



e) 1000°C-2 hr-OQ-600°C-1 hr MSS at 50X

f) 1000°C-3 hr-OQ-600°C-1 hr MSS at 50X



g) 1000°C-4 hr-OQ-600°C-1 hr MSS at 100X

Fig.5.1 Microstructure of as received and heat treated 13/4 martensitic stainless steel

5.1.2 EFFECT OF HEAT TREATMENT ON MECHANICAL PROPERTIES

Table 5.1 demonstrates the effects of tempering time and the austenitization of temperature on the mechanical properties. This is systematically plotted in the fig 5.1-5.5. The data table clearly illustrates that when compared to the as received material, the heat treatment improves all the properties being mechanical in nature.

The ultimate tensile strength (UTS) value is 1138 MPa in as received condition. In the heat treated samples or specimens, it has been detected that the UTS varies in the range between 733 – 1038MPa. The results certify that the strength shall be reduced by giving the heat

treatments to the as received materials. In terms of percentage elongations, the ductility can be predicted. The heat treatments given to the cast material improves the ductility consistently. In the as received condition, the percentage elongation is 6.05% whereas in case of samples being heat treated, it shall vary between 6.00 – 8.80%

The toughness also varies in the same dialect. Therefore, in the as received condition, the toughness is 4.3 Kg-m. whereas it varies between 5.9 - 8.9 Kg-m. in case of samples being heat treated. Heat treatments given to the as received material improves the intensity of toughness. The hardness value in case of as received is 380 VHN, whereas it varies between 230 – 324 VHN in case of heat treated samples. This can be observed from the given results that the hardness reduces due to the heat treatments given to the as received materials.

Table 5.1 Mechanical properties of as received and heat treated 13/4 martensitic stainless-steel samples.

SPECIMEN SPECIFICATIONS	TENSILE TEST		CHARPY IMPACT TEST	HARDNESS TEST
	UTS (MPa)	PERCENTAGE ELONGATION	TOUGHNESS (Kg-M)	VHN P = 10 Kg
As received	1138	6.05	4.3	380
950°C-2 hr-OQ-600°C-1 hr	844	6.33	6.0	279
950°C-3 hr-OQ-600°C-1 hr	927	7.75	6.9	230
950°C-4 hr-OQ-600°C-1 hr	733	6.00	8.9	236
1000°C-2 hr-OQ-600°C-1 hr	871	6.30	6.4	314
1000°C-3 hr-OQ-600°C-1 hr	946	7.60	6.2	284
1000°C-4 hr-OQ-600°C-1 hr	1027	8.80	5.9	324

As the mechanism of erosion is linked to the mechanical properties of the materials, this makes the evaluation of mechanical properties essential.

Firstly, the tensile strength increase and thereafter it decrease with the soaking time. This is clarified from the fig 5.2-5.5. These are plotted at the different austenitizing temperature between the soaking time and the mechanical properties. With the decreasing soaking duration, the toughness and the percentage elongation also marginally increase. The opposite is the behaviour in case with hardness of material. Firstly, the hardness decrease and subsequently it increase after 2 hours of soaking duration. But the austenitizing temperature which is 950°C decrease furthermore after 2 hours of soaking duration.

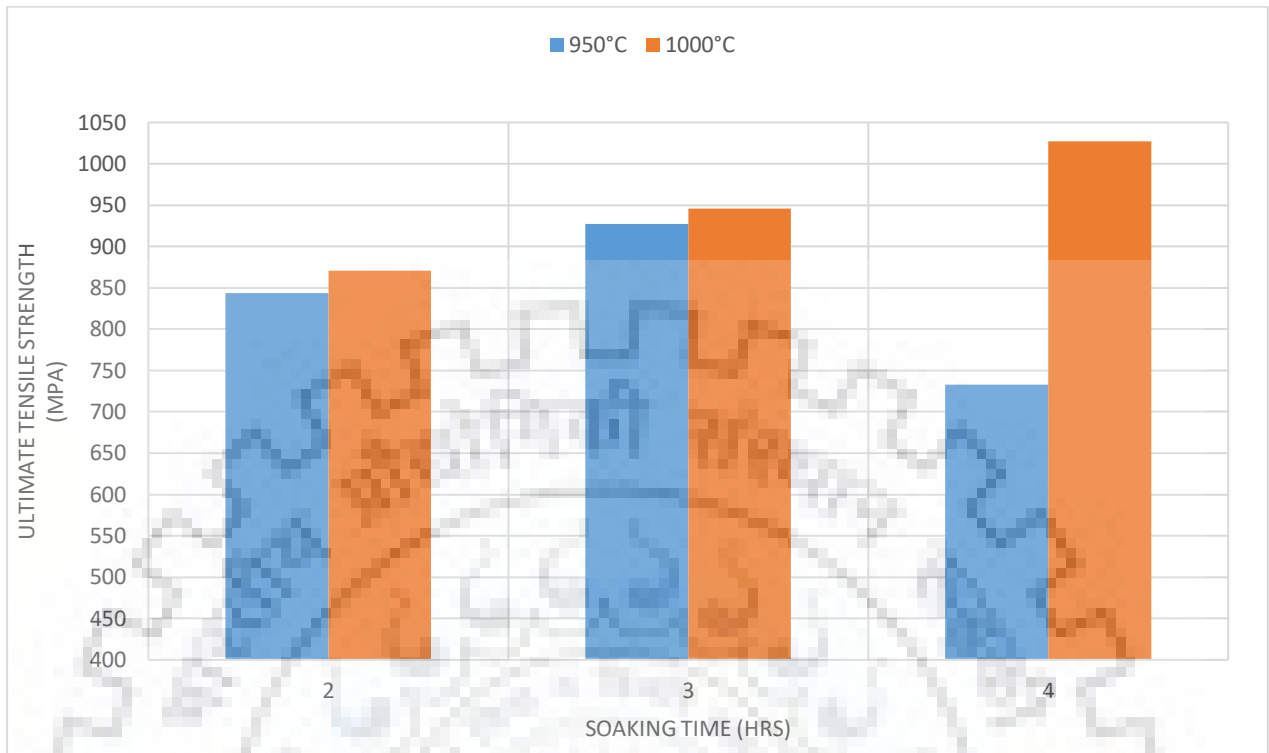


Fig 5.2 Effect of soaking time on UTS at different austenitizing temperature.

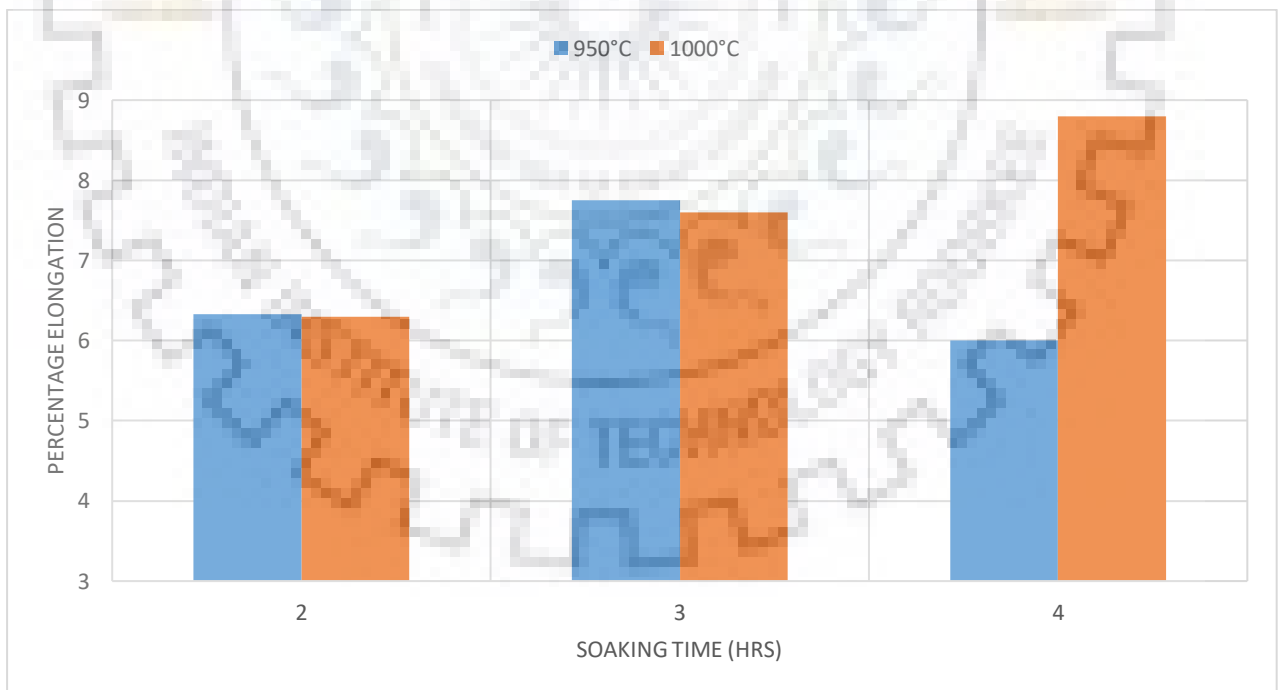


Fig 5.3 Effect of soaking time on % elongation at different austenitizing temperature for 13/4 martensitic stainless steel

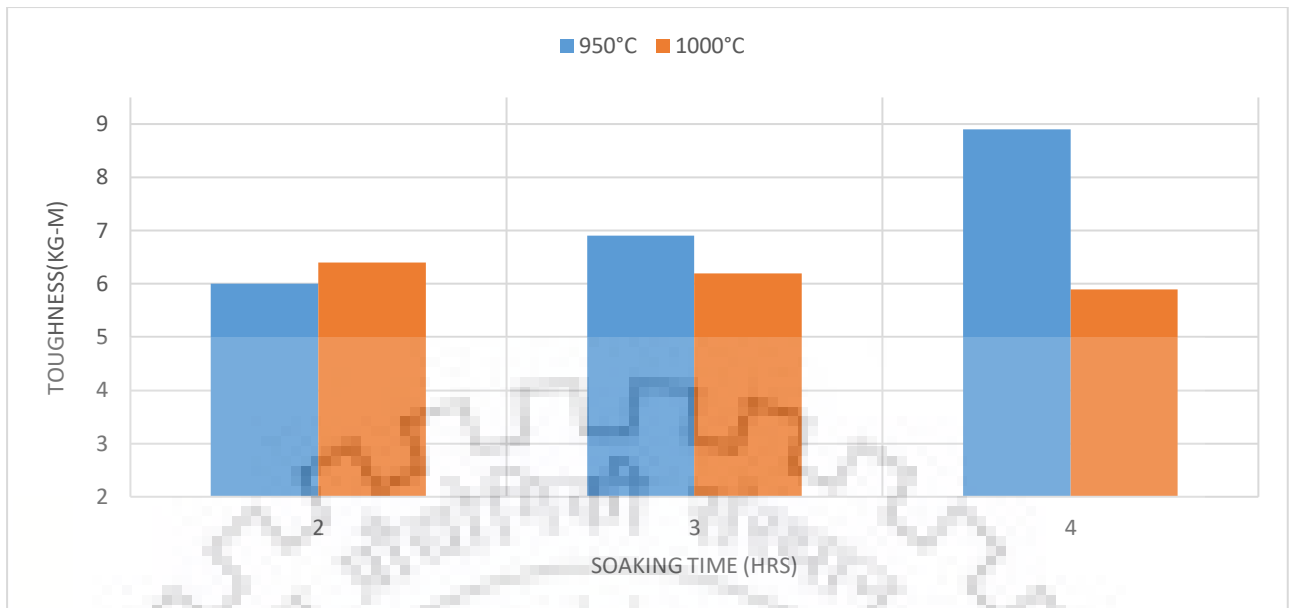


Fig. 5.4 Effect of soaking time on toughness at different austenitizing temperature for 13/4 martensitic stainless steel

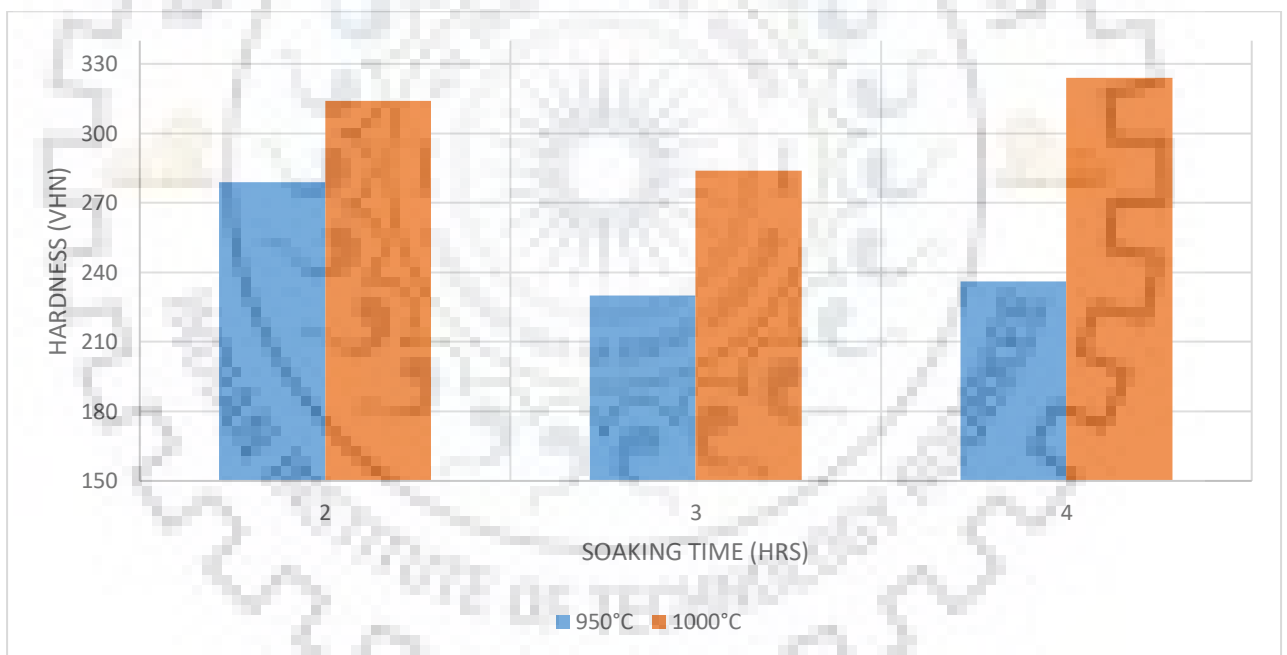


Fig. 5.5 Effect of soaking time on hardness at different austenitizing temperature for 13/4 martensitic stainless steel

5.2 EROSION TEST RESULTS

The studies have revealed the erosion behaviour of the as received material and samples that has been heat treated. Table 5.2 comprises of the initial dimensions and the weight of samples of various categories. the results are recorded of the experiments done in order to discover the weight loss and the cumulative weight loss in slurry erosion tester at 30° angle. The exposure time for such experiment is 4 hours. In the table 5.13, cumulative weight loss calculation is presented. This weight loss is presented in terms of weight loss per unit surface area.

5.2.1 EFFECT OF HEAT TREATMENT ON EROSIVE WEAR RATE

Table 5.3-5.4 acknowledges the soaking duration on the erosion of material at 30° angle and the results or effects of austenitizing temperature based on the experiments done on the erosion. It has been understood that in case of 30°angle, 0.142 gm shall be weight loss in total of as received material. Moreover, the weight loss in total shall vary between 0.080 – 0.165 gm in heat treated conditions.

The table demonstrates the cumulative weight loss in terms in terms of weight loss per unit surface area that shall be calculated. It was observed after 16 hours that in case of 30°impingement angle, for the as received material, the weight loss in total shall be

$38.40 \times 10^{-5} \text{ gm/mm}^2$. Moreover, the total weight loss shall vary between $20.34 \times 10^{-5} - 40.73 \times 10^{-5} \text{ gm/mm}^2$ in the heat-treated conditions.

Table 5.2 initial Data of Erosion Test samples of 13/4 martensitic stainless steel

Specimen Specification	Length 'l' (mm)	Breadth 'b' (mm)	Thickness 't' (mm)	Weight (gm)	Surface area (mm²) =2[(l x t)]+ [l x b]
As received (a) 30°	23.89	9.52	2.98	5.334	369.817
950°C-2 hr-OQ-600°C-1 hr (a) 30°	24.47	9.35	3.36	6.247	393.234
950°C-3 hr-OQ-600°C-1 hr (a) 30°	23.89	9.56	2.98	6.524	385.924
950°C-4 hr-OQ-600°C-1 hr (a) 30°	24.28	8.76	3.34	5.682	374.883
1000°C-2 hr-OQ-600°C-1 hr (a) 30°	24.58	9.56	3.18	5.414	391.312
1000°C-3 hr-OQ-600°C-1 hr (a) 30°	24.87	9.49	3.50	6.630	410.106
1000°C-4 hr-OQ-600°C-1 hr (a) 30°	24.78	8.98	3.04	4.935	373.189

Table 5.3 Erosion Data of as received and different heat treated 13/4 martensitic stainless steel in Erosion Test at 30° angle

SPECIMEN SPECIFICATIONS	As Received		950°C-2hr		950°C-3hr		950°C-4hr	
	Wt. loss (gm)	Cum. loss (gm)	Wt. loss (gm)	Cum. loss (gm)	Wt. loss (gm)	Cum. loss (gm)	Wt. loss (gm)	Cum. loss (gm)
0	0	0	0	0	0	0	0	0
4	0.013	0.013	0.024	0.024	0.024	0.024	0.034	0.034
8	0.061	0.074	0.021	0.045	0.027	0.051	0.029	0.063
12	0.041	0.115	0.015	0.060	0.022	0.073	0.026	0.089
16	0.027	0.142	0.020	0.080	0.023	0.096	0.036	0.125
Total weight loss (gm)	0.142		0.080		0.096		0.125	

SPECIMEN SPECIFICATIONS	1000°C-2hr		1000°C-3hr		1000°C-4hr	
	Wt. loss (gm)	Cum. loss (gm)	Wt. loss (gm)	Cum. loss (gm)	Wt. loss (gm)	Cum. loss (gm)
0	0	0	0	0	0	0
4	0.014	0.014	0.028	0.028	0.028	0.028
8	0.049	0.063	0.036	0.065	0.055	0.083
12	0.051	0.114	0.026	0.091	0.042	0.125
16	0.026	0.140	0.026	0.117	0.027	0.152
Total weight loss (gm)	0.140		0.117		0.152	

Table 5.4 Cumulative weight loss in terms of weight loss per unit surface area of 13/4 martensitic stainless steel

SPECIMEN SPECIFICATION	CUMULATIVE WEIGHT LOSS (10^{-5} gm/mm ²)				
	0 hr	4 hr	8 hr	12 hr	16 hr
As Received (a) 30°	0	3.52	20.00	31.09	38.40
950°C-2 hr-OQ-600°C-1 hr (a) 30°	0	6.10	11.44	15.26	20.34
950°C-3 hr-OQ-600°C-1 hr (a) 30°	0	6.21	13.22	18.92	24.88
950°C-4 hr-OQ-600°C-1 hr (a) 30°	0	9.07	16.81	23.74	33.34
1000°C-2 hr-OQ-600°C-1 hr (a) 30°	0	3.57	16.09	29.13	35.77
1000°C-3 hr-OQ-600°C-1 hr (a) 30°	0	6.83	15.85	22.19	28.53
1000°C-4 hr-OQ-600°C-1 hr (a) 30°	0	7.50	22.24	33.50	40.73

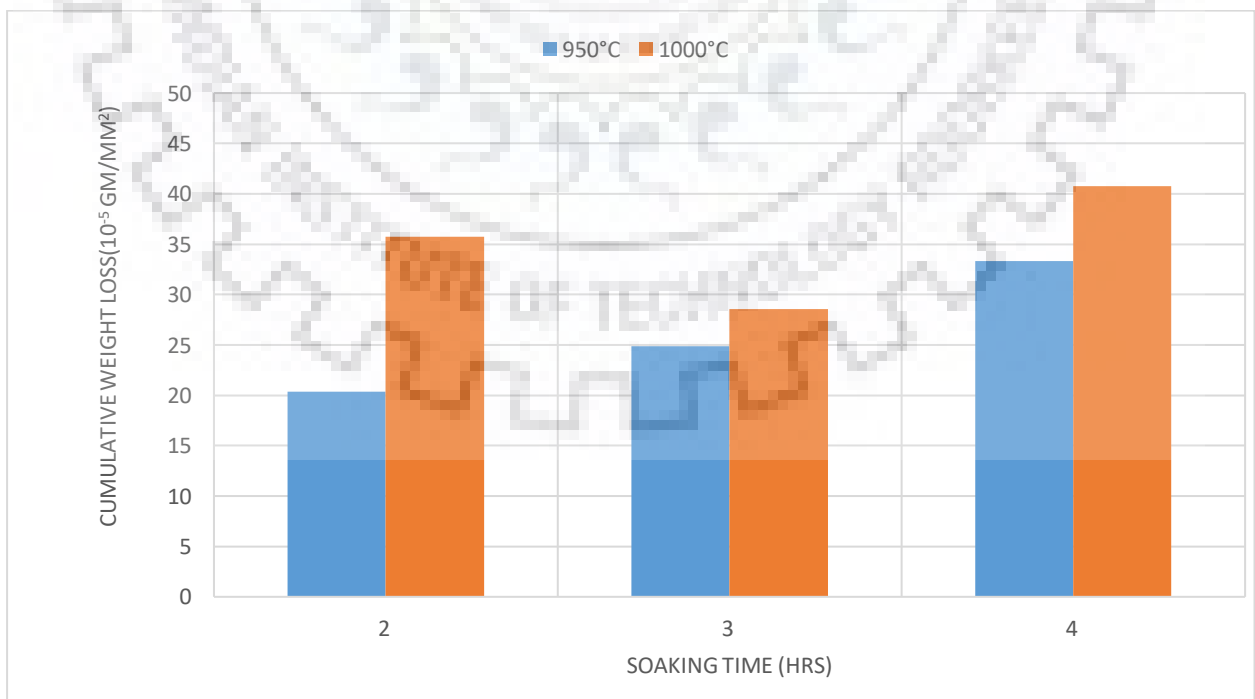


Fig. 5.6 Effect of soaking time on Total weight loss for 13/4 martensitic stainless steel

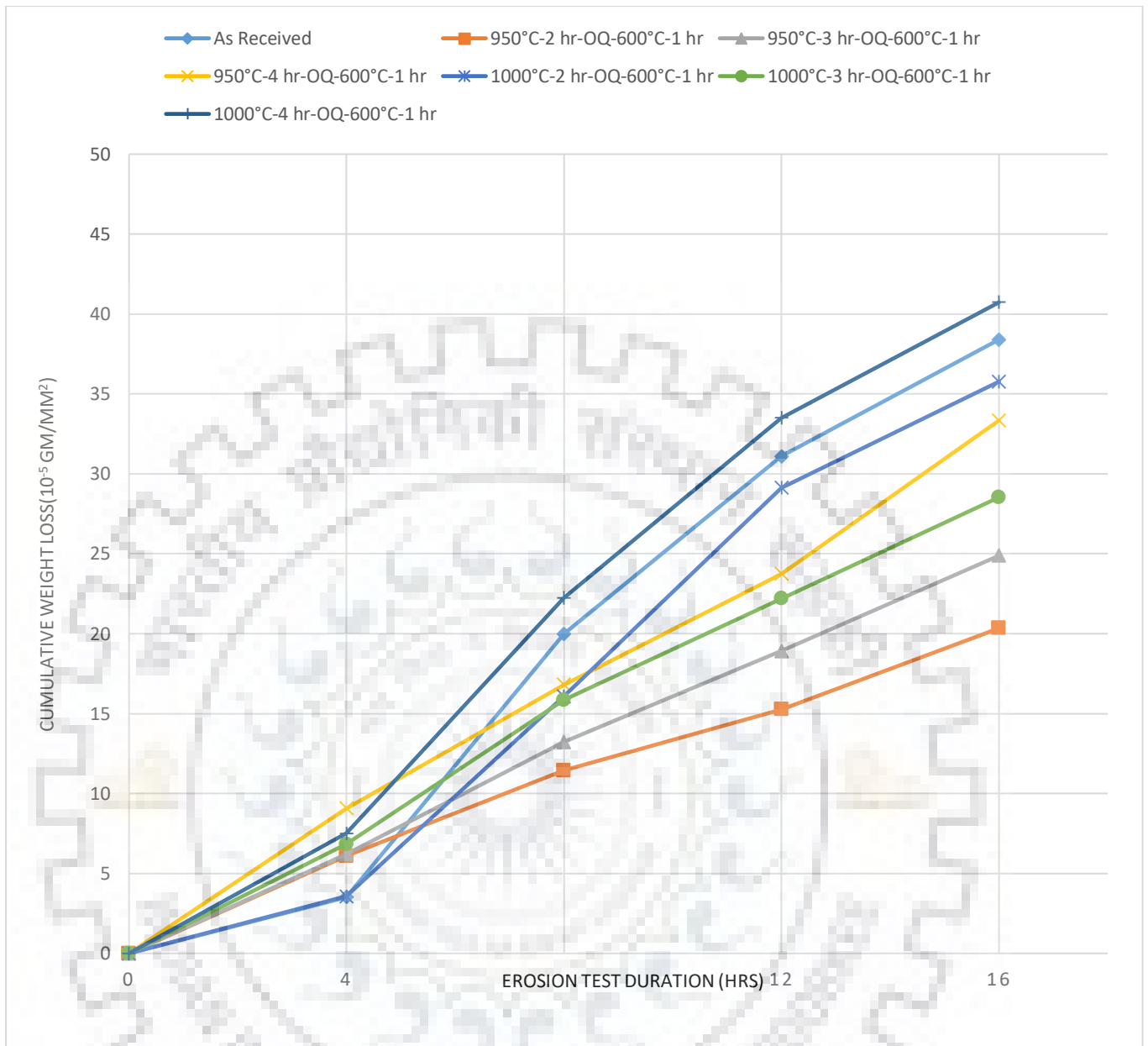


Fig. 5.7 Cumulative weight loss at Impingement angle: 30° for 13/4 martensitic stainless steel

The above given figure made at 30° angle, portrays the cumulative weight loss and duration of erosion test, the erosive wear rate of heat treated samples are comparatively lower than the erosive wear rate of as received sample. The minimum erosion occurs for heat treatment at 950°C-2 hr-OQ-600°C-1 hr followed by 950°C-4 hr-OQ-600°C-1 hr, 1000°C-3 hr-OQ-600°C-1 hr, respectively.

5.2.2 EFFECT OF MECHANICAL PROPERTIES ON EROSIVE WEAR RATE

The table clarifies the effect of mechanical properties on erosive nature. The given plots differentiates between several mechanical properties and weight loss in total.

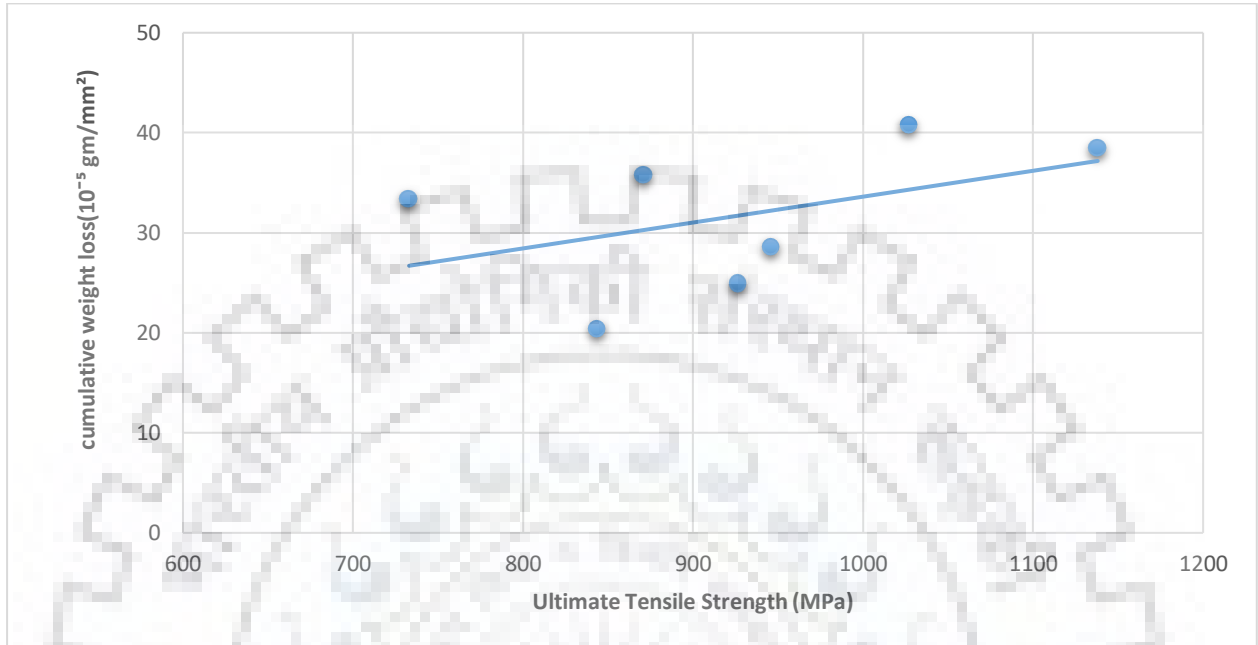


Fig. 5.8 Effect of UTS on Total weight at 30° angle for 13/4 martensitic stainless steel

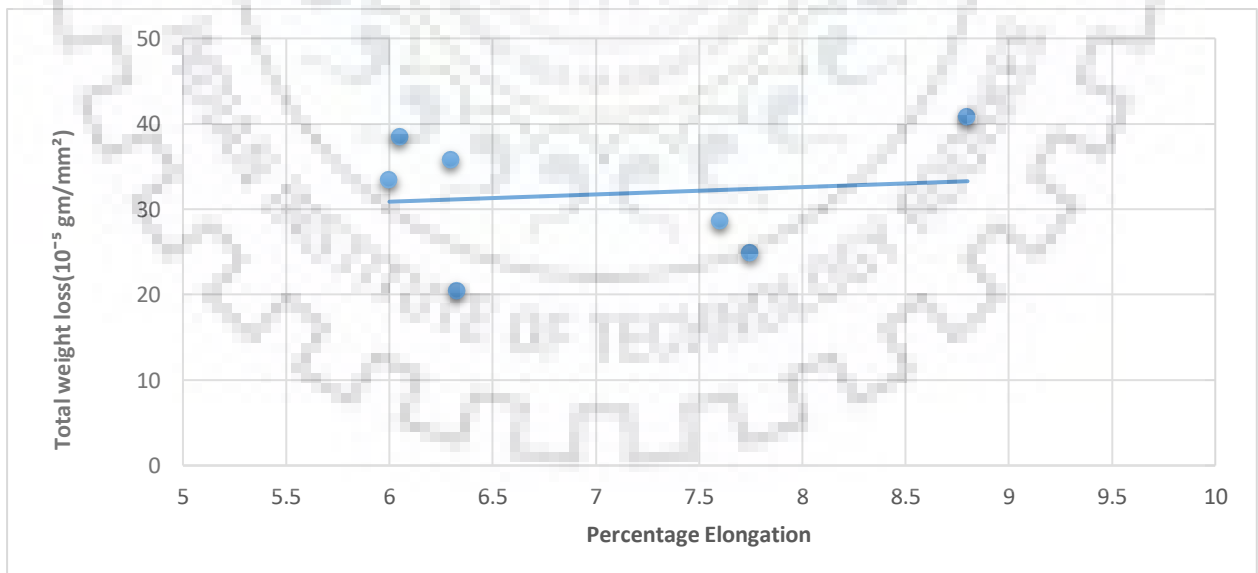


Fig. 5.9 Effect of % Elongation on Total weight at 30° angler for 13/4 martensitic stainless steel

From the above shown diagrams of UTS and loss of weight in total, it is clarified that with the increase in UTS, the erosive wear also increases marginally. Also, the above plotted diagram demonstrates that with the increase in ductility known as percentage elongation, the erosive wear marginally decrease simultaneously. In case of toughness in nature, the erosive wear decrease with the increasing toughness. As of the effect in case of UTS, the hardness shows the similar nature as of UTS. With the increasing hardness, the erosive wear increase simultaneously.

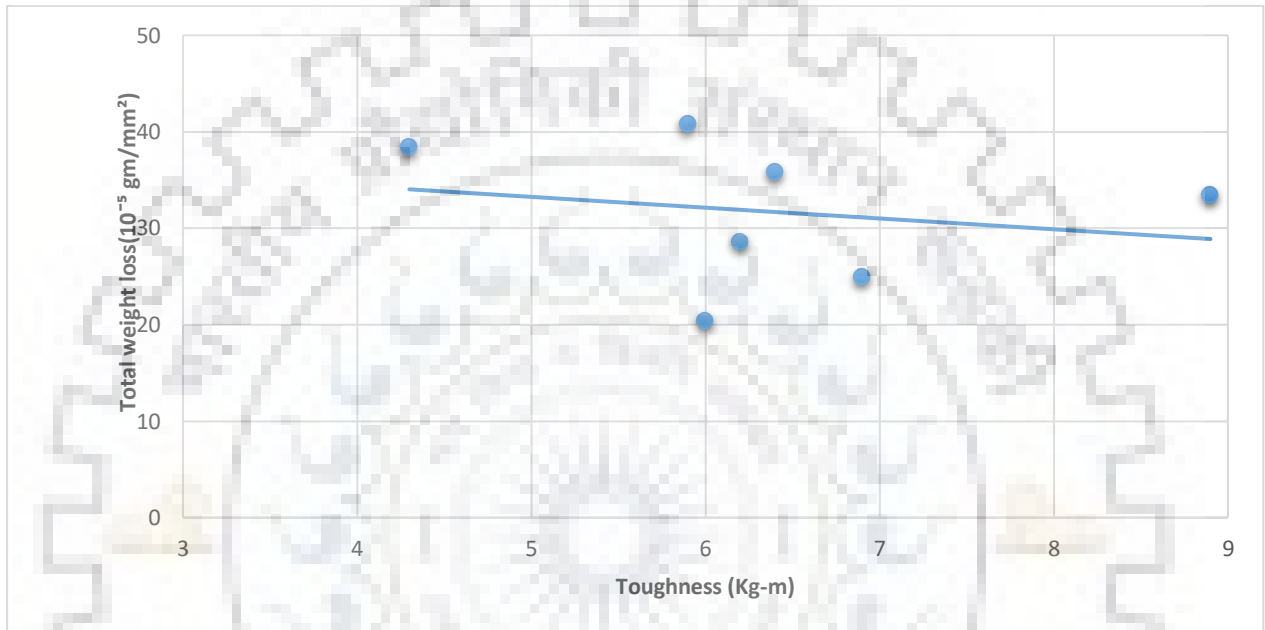


Fig 5.10 Effect of Toughness on Total weight at 30° angle for 13/4 martensitic stainless steel

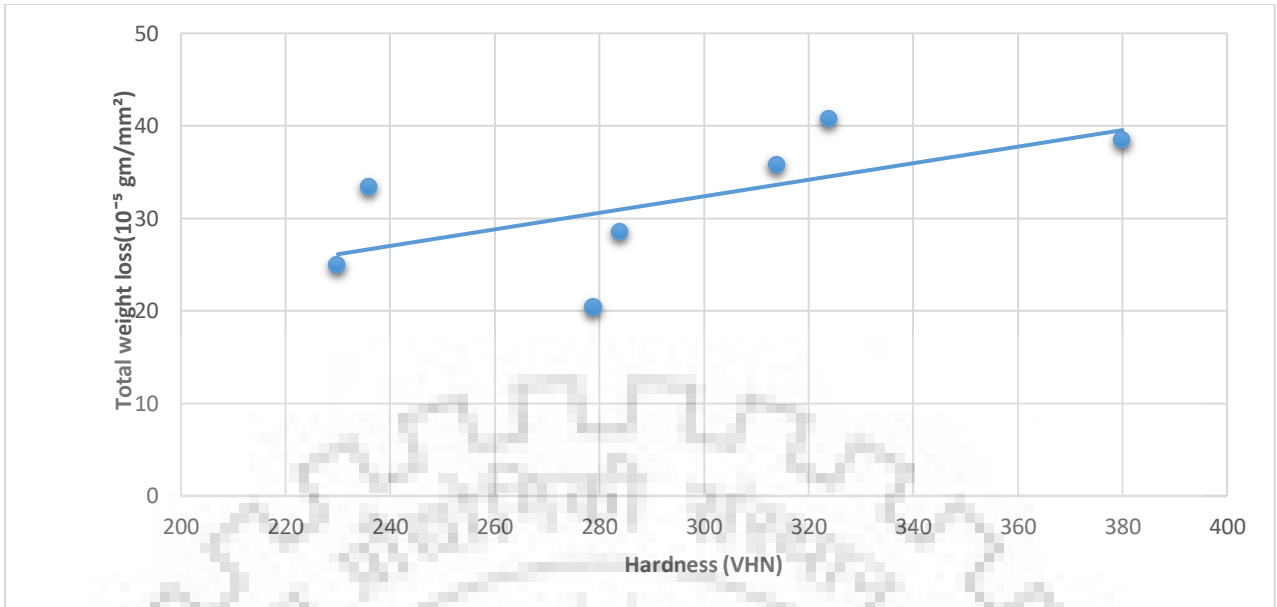


Fig. 5.11 Effect of Hardness on Total weight at 30° angle for 13/4 martensitic stainless steel

The above plotted diagram shows that the similar effects are shown by hardness and UTS with the total loss in weight. It is also observed that the erosive wear is inversely proportional to the toughness and the percentage elongation or ductility. The similar effects are shown by the ductility and toughness.

5.2.3 COMPARISON BETWEEN AS RECEIVED AND HEAT TREATED MATERIALS WEAR

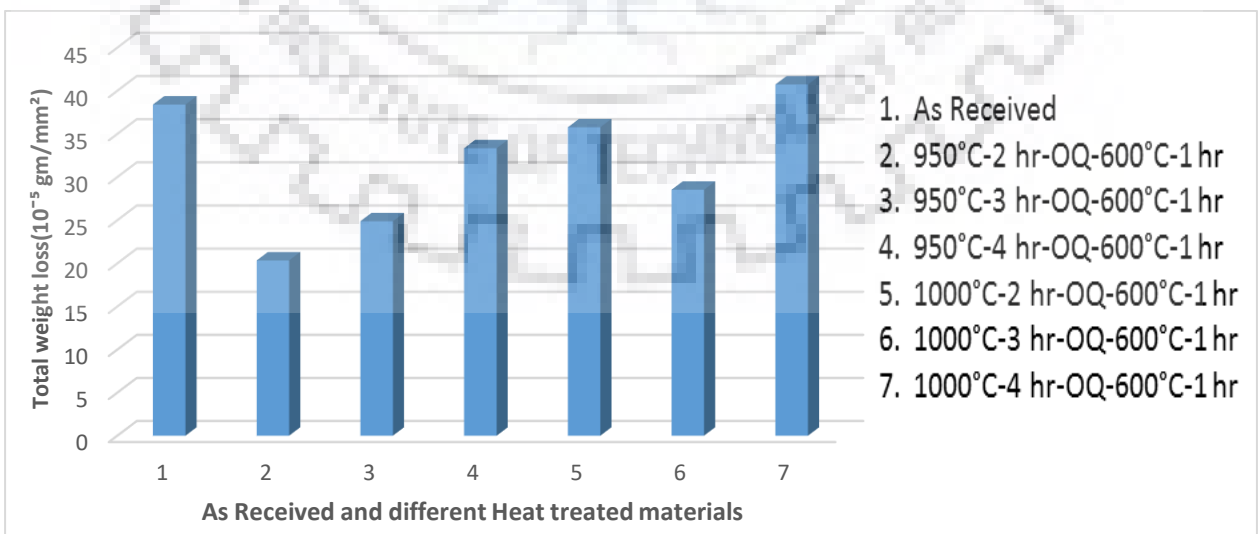


Fig. 5.12 Comparison between As received and heat treated materials

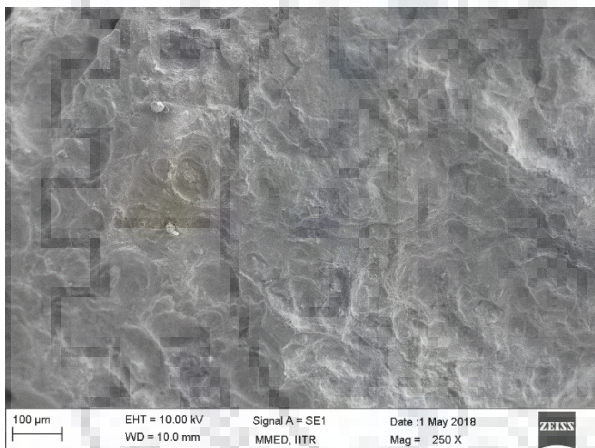
In seen the bar chart heat treated specimen at 30° angle has high Erosion wear loss as compared to As received materials. . The minimum erosion occurs for heat treatment at 950°C-2 hr-OQ-600°C-1 hr followed by 950°C-3 hr-OQ-600°C-1 hr, 1000°C-3 hr-OQ-600°C-1 hr, 950°C-4 hr-OQ-600°C-1 hr, respectively.

5.3 SCANNING ELECTRON MICROSCOPIC (SEM) STUDIES

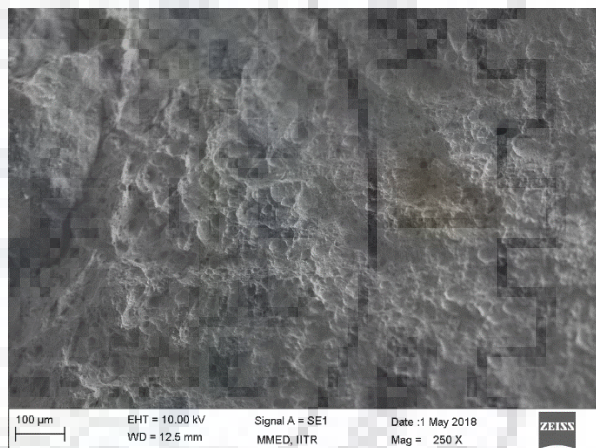
The scanning electron microscopic observations has been made from the surface of As received and heat treated specimen as shown in the figure

5.3.1 FRACTURED SURFACE

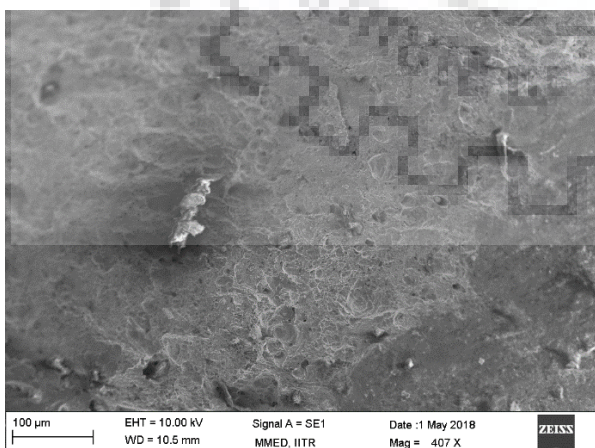
For gaining an insight into the behaviour of a fracture, the fractured surface studies has been done under electron microscope for both charpy and tensile specimens.



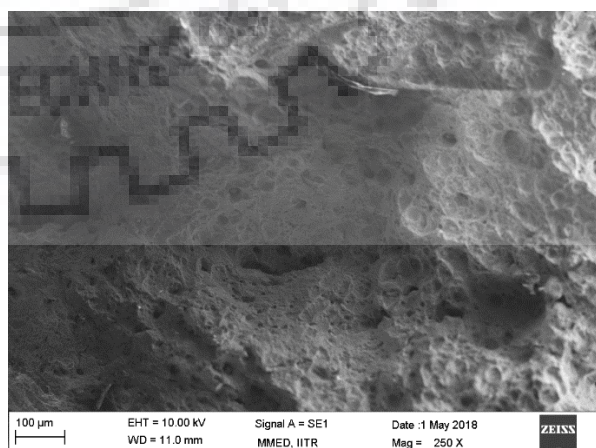
a) As received 13/4 MSS at 250X



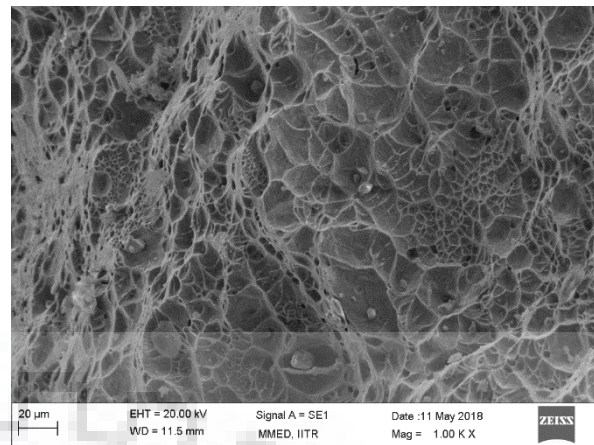
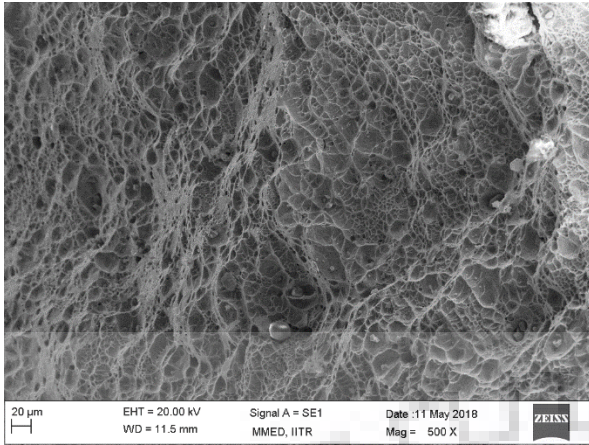
b) 950°C-2 hr-OQ-600°C-1 hr MSS at 250X



c) 950°C-3 hr-OQ-600°C-1 hr MSS at 407X



d) 950°C-4 hr-OQ-600°C-1 hr MSS at 250X



e) 1000°C-2 hr-OQ-600°C-1 hr MSS at 500X

f) 1000°C-3 hr-OQ-600°C-1 hr MSS at 1000X



g) 1000°C-4 hr-OQ-600°C-1 hr MSS at 250X

Fig. 5.13 Fractured surface of Tensile specimens of 13/4 martensitic stainless steel

The figure given above clarifies that the majority of the facets and cleavage fracture being one of the properties of the brittle fracture is shown by the as received specimen under tension. Also, on the other side, the specimen which shows the erosive wear in less amount as compared to all the other samples is 950°C-2hr-OQ-600°C-1hr. this specimen have got characteristic similar to the ductile fracture such that it has got many dimples. The induction in ductility due to the loss of hardness is the result of the austenitization provided to the specimens.

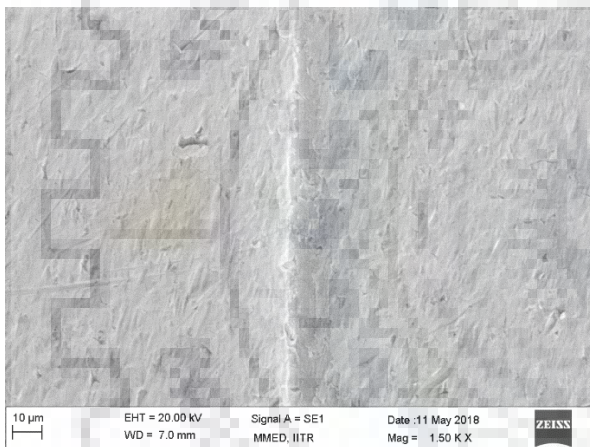
It can be observed from the above given figure that the similar properties are shown by charpy test in the as received specimen as of the tensile test known as brittle fracture. The other two specimens that shows the erosive wear in nominal amount are 950°C-2hr-OQ-600°C-1hr and 950°C-4hr-OQ-600°C-1hr and it has got the properties similar to that of a ductile fracture. Moreover, 1000°C-2hr-OQ-600°C-1hr is the specimen that has revealed the

maximum amount of erosive wear. The reasons are clarified above in a systematic manner already. The dimples are tends to be formed majorly in the tensile test as compared to the charpy test.

5.3.2 ERODED SURFACE

From the surface of the as received and heat treated samples exhibiting the weight loss in nominal amount during erosion after erosion test duration of 16 hours. The details of the mechanism of erosion is being provided by the SEM studies.

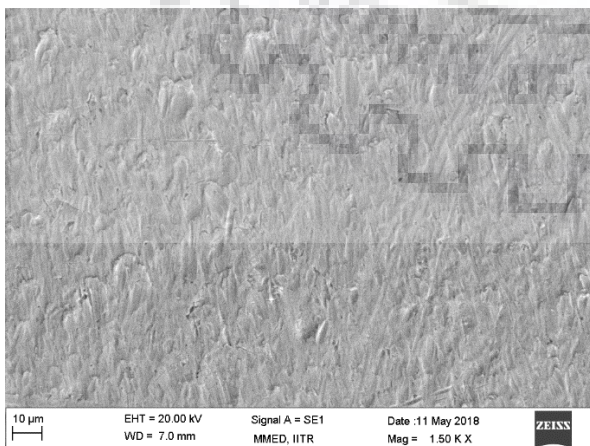
In the fig.5.14, the surface morphology of As received and heat treated materials after the test of erosion is shown. Also, the morphology after the test of erosion is shown in the subsequent figure.



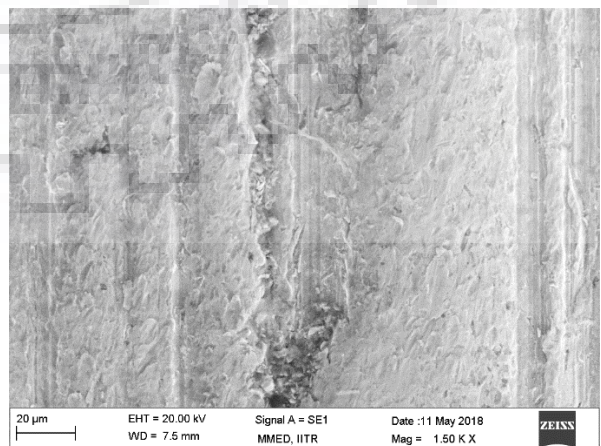
a) As received 13/4 MSS at 1500X



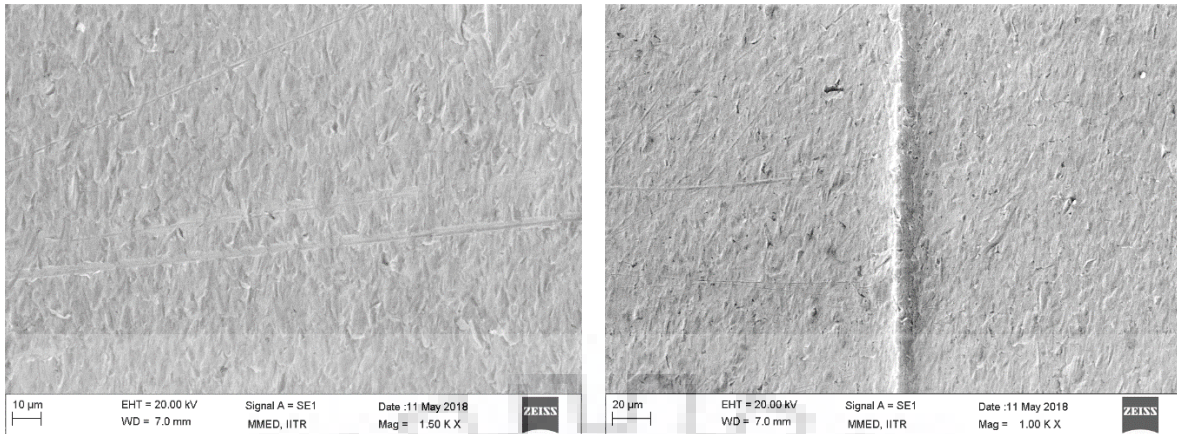
b) 950°C-2 hr-OQ-600°C-1 hr MSS at 1500X



c) 950°C-3 hr-OQ-600°C-1 hr MSS at 1500X



d) 950°C-4 hr-OQ-600°C-1 hr MSS at 1500X



e) 1000°C-2 hr-OQ-600°C-1 hr MSS at 1500X f) 1000°C-3 hr-OQ-600°C-1 hr MSS at 1000X



g) 1000°C-4 hr-OQ-600°C-1 hr MSS at 1500X

Fig. 5.14 Surface morphology of 13/4 martensitic stainless steel at impingement angle 30°

A couple of different mechanisms of erosion naming cutting and ploughing were demonstrated by SEM observation of eroded surfaces. Plastic deformation is caused firstly later on followed by the removal of surface. At the bank of the grooves, the ridges were formed. after subsequent impact of the silt. In the eroded surface, the cutting scars are also there. The morphology is similar in case of all eroded surface in heat treated and as received specimens. Therefore, the mechanism of removal of material is also found to be similar.

At the low and high angles, the mechanism of material removal is found to be distinct. At angle of 30°, during particles impact surface at low angles, in the wear scars there is directionality. During this, the ridges were pushed in front of them alongwith gouging the particles at surface being known as Chips.

5.4 DISCUSSION

The microstructures are analysed and discussed pertaining to the effect of various heat treatments on properties being mechanical in nature. For understanding the influence of austenitizing temperature and tempering duration on the various properties of 13/4 martensitic stainless steel, optical and SEM studies is being done. The mechanism of erosion along with the later effects on behaviour of erosion are discussed.

5.4.1 STRUCTURE PROPERTY CORRELATION

During the investigation, the weight loss of the steel in erosion, the microstructures play an effective role for controlling such hazard along with other properties. Precipitation of carbides, retained austenite along with the balance of martensitic are all included in the principle microstructure.

In The behaviour of erosion and other mechanical properties, carbides play an effective role. The carbide precipitates corresponding to the composition M_3C , M_7C_3 and $M_{23}C_6$ in 13/4 martensitic stainless steel. During austenitization, following changes occur:

- i. The size of austenite grain size increases with the increase in austenitizing temperature
- ii. At the time of soaking, carbides and alloying elements gets dissolved.

By increasing the austenitizing temperature, there shall be the enhancement in the extent of dissolution of carbides and alloying elements in alloy. There shall be increment in the tendency of formation of martensite due to the size of coarse grain austenite and the composition being homogeneous. There shall occur the undesirable changes in several other properties like loss in the ductility or the brittleness being increased. With the prior increase in size of austenite grain, the retained austenite increases at the same time. Carbon is precipitated as particles of carbide being very fine in consistency at the time of tempering. These are distributed in uniform nature in the matrix. The process of tempering produces relatively finer carbide of similar type in the grain cores along with the depletion in chromium as per Brezina et. al. [49]. This depletion occurs at both at the boundary of grain as well as within the grain area.

There is a strong rise in the amount of precipitation with fine M_6C carbides. Next to this, $M_{23}C_6$ carbides in the quenched and tempered martensitic stainless steel being already precipitated in

huge number of crystal cores. This is observed by Heimann et. al. [50] and Gumpel [51]. Many authors have deeply investigated the formation of carbides in 13/4 martensitic stainless steel [52, 53].

The particles of carbide are generally sub microscopic in starting that is very small in size. There are distinct changes observed while tempering at the temperature of 600°C. These are as follows:

- (i) A fine acicular structure of grain is developed with the rapid softening in the starting where dislocation of cell walls within the annihilation of laths of martensite.
- (ii) At the boundaries, by the pinning actions of carbides of alloy, the recrystallization process is inhibited.

The process of growth and coalescence results in the growth of carbides to size of microscopic in nature with the continuous heating. In the present studies, at the high temperature of tempering, this coalescence is observed to be more pronounced. Keeping aside the carbides, formation of austenite also affects during various heat treatments. At the tempering range of 600°C, the 13/4 martensitic stainless steel owes its best properties such that the finely dispersed austenite cannot be detected by optical microscope. Process of segregation makes this finely dispersed austenite stable.

Untempered martensite of as received microstructure shown by the figure 5.1(a) provides the tensile strength of maximum range. Austenitizing at temperature of 950°C and 1000°C for different soaking duration alongwith tempering at 600 °C decreases the value. Fine martensite lath in little packets that increases the tensile strength to an optimum level is revealed by the as received microstructure. The initial process of yielding is made difficult due to the migration of atoms of carbon to the boundaries of packet. Fig. 5.1(b-g) reveals the tempered martensite by the microstructures in heat treated samples. As the degree of hardening increases, the tensile strength increases similarly with the austenitizing temperature.

Ductility deteriorates (6.05%) in the as received structure with the presence of the coarse carbides along the boundaries of grains. At all austenitizing temperature, percentage elongation increases. In the present study, the prior austenite grain coarsening is responsible for the decrease in ductility at 950 C 4 hr heat treatment. Because grain coarsening results in coarse lath formation. With the increase in soaking time, amount of retained austenite increases. The

carbide formation is easier from austenite. Therefore, in 4 hours, the larger size of carbides is obtained leading to minimum ductility.

The minimum value of ductility was observed to be 6.30% at austenitization at 1000°C and 2 hours soaking duration.

A thorough investigation has been done on the influence of micro constituents on UTS. With the increase in soaking duration, UTS value also decreases. Prior austenite grains are coarser at the temperature of 1000° and amount of retained austenite increases gradually with soaking duration. Maximum tensile strength is observed at 1000°C austenitizing temperature corresponding to soaking duration of 4 hours exhibited from the combination of these two factors

5.4.2 EFFECT OF MICROSTRUCTURE ON EROSION BEHAVIOUR

It was observed in this study that the basic constituent or matrix is martensite. Erosion resistance is also benefitted from the formation of bainite. The reduction of erosion is resulted from the existence of retained austenite. Erosion studies are being provided by Salik et. al. [54] on AISI steel in the normalised, water quenched and tempered condition. The erosion resistance is more likely to get affected from the metallurgical changes as compared to the hardness. Therefore, under the condition of erosion, behaviour of steel to be seen jointly with microstructure and properties that are mechanical in nature. It would be incomplete to correlate the data of erosion only on the basis of mechanical properties.

Erosion tend to increase with the presence of carbide in the martensitic matrix, observed from the study done presently. Due to various effects, these carbides promote the erosion. During the impact of silt particles being opened up at these surface, interface of carbides is weak on them. Due to the hardness being so high, carbides also cause plastic flow to be inhomogeneous. From the retained austenite whose quantity increases with the soaking duration, the precipitation of carbide takes place easily. Therefore, at the soaking duration of 4 hours at the temperature of 950°C, the erosion occurs in very high intensity. Strain

localization occurs causing the cracking. These are generally found near carbides resulting in the enhancement of erosion of material. Carbide fracture is also prone to be caused due to the associated high stresses.

The weight loss in total in case of slurry erosion is comparatively lower than that of as received material in all heat treated condition except the case of heat treatment for the soaking duration of 4 hours at the temperature of 1000°C. Even after the soaking time of 4 hours at the temperature of 1000°C, the complete dissolution of carbides shall not occur. During the subsequent tempering, the particles of carbide shall provide the nucleus prepared for the growth of carbide. With the increasing duration of soaking, amount of retained austenite increases leading to the precipitation of carbides. Thus, at the soaking duration of 4 hours at the temperature of 1000°C, the loss in weight is highest.

At 2 hours of soaking duration at 950°C, austenitizing temperature, the weight loss is reported in the minimum amount. With the increase in soaking duration, at this temperature, homogeneity and the size of prior austenite grain increases in the same intensity. These all results in the material, the formation of lath of martensite. As we know that with the increase in soaking duration, the retained austenite also increases. During erosion test by induced transformation, it is transformed partially to martensite. The softness in the material is caused by the high amount of retained austenite that is not desirable in such case. The erosion caused shall be high in case of coarser martensitic laths along with high amount of retained austenite as compared to the less quantity of retained austenite with fine martensite lath. As compared to the higher soaking duration, the amount of precipitation of carbides is high than the lower one. The factors mentioned above has a condition optimum in nature corresponding to 950°C-2 hours.

At 950°C, the loss in weight is minimum as compared to the loss in weight at the temperature of 1000°C. The amount of weight loss depends on the precipitation extent, amount of retained austenite and the size of packet of lath martensite. At 1000°C, the size of lath martensite packet is coarse due to the partial dissolution of carbides and grain coarsening at the time of austenitizing, precipitation of carbides is comparatively of high intensity. Nevertheless, packet of lath martensite will still be fine at the temperature of 950°C but there would be a slight increment in the precipitation of carbides. This will be due to the dissolution of carbides completely at the time of austenitization. Thus, at the high heat treatment temperature, the

weight loss would be high. The loss in weight shall be the minimum at the heat treating temperature of 950°C at optimum values of such parameters.

5.4.3 EFFECT OF MECHANICAL PROPERTIES ON EROSION BEHAVIOUR

One of the important parameter that affects the erosion are the mechanical properties of the target material. Taking example of the particles of silt that impacts on the surface of target material thereby causing erosion. The plastic flow of the target material by impact of silt laden water. This is supports by ductility. At the time when the particles of silt causes tensile stresses in the material, UTS comes in the reign. Capacity of deformation of plastic is reduced by the hardness that further facilitates in the reduction of scratching and ploughing. Impact damages are reduced with impact strength. Therefore, the erosion behaviour of material is greatly affected by the mechanical properties.

It is observed in the present study that ductility of the material greatly affects the loss in weight of the target material. There is reduction in loss of weight due to the increasing ductility of target material. This trend is being observed in the studies. Also, increase in UTS value increases the loss in weight. The weight loss decreases with the increasing toughness. Furthermore, hardness increases the weight loss.

There is no individual mechanical property that controls the behaviour of erosion of steel totally. This is concluded from the studies done recently. This fact can be truly verified from the case when the silt impacts the water slurry on the material, there is no single factor that affects erosion individually. Several mechanical properties affects the mechanism of erosion. Along with the mechanical properties, the micro constituents also play an effective role in affecting the erosion. Thus, there is need of a critical analysis on the problem of erosion as this issue is a complex one and affects many industries.

Erosion relates to the hardening, ductility and rate of work. This is revealed by many investigators [8]. There is a great role of ductility as the hardness has the tendency of localization of strains and development of cracks. 13/4 martensitic stainless steel possess great hardness of high level. The treatment results in improving the ductility thereby reduces the erosion sharply. It is observed that the behaviour of erosion is not altered substantially with the change in ductility at the levels of high ductility. Therefore the best option for the high erosion resistance is the material of very high hardness with an adequate amount of ductility. Due to

the strain localization, the stresses are dissipated in such material having the ductility. Here, the chances of formation of cracks are deterred.

The formation of ploughs and shear lips is reduced as there is less amount of ductility possessed by the material. Mechanism of erosion in the steel is related to the deformation of plastic and shear lips and ploughs formation. These have been revealed in the SEM studies. There is an inverse relation between hardness and the ductility. The former increases with the reduction in amount of the latter. Hutching [55] predicted that there is an innate expectation that both high intensity of hardness as well as a high ductility is adequate for the resistance of erosion keeping in mind the material displacement and plasticity. A deep study has been conducted by Naim et.al. [56] on the behaviour of erosion on a steel of 18% Ni maraging. The hardness of this steel could be changed over a limited range with almost no change in the ductility. It is observed that as the hardness increases the erosion also increases at the ductility remaining constant. These results conflict the observations of Finnie et. al.[32]. Finnie concludes that there is reduction in erosion with the increase in hardness. In the constant strength ranges, there is an increase in the rate of erosion when there is reduction in the ductility. This is concluded by Naim. The point to be kept in mind is that the measurement of ductility in the test of quasi static tensile is distinct in comparison from ductility relating at the very high rate of strain of impact of the particles.



CONCLUSIONS

6.1 CONCLUSIONS

The thorough studies are conducted on the effect of treatment of heat on the mechanical properties, structures and behaviour of silt erosion on materials of 13/4 martensitic stainless steel. Austenitization at temperature of 950°C and 1000°C at the intervals of 2 hours, 3hours and 4 hours respectively are involved by heat treatments. This shall be followed with tempering being provided at 600°C for the duration of an hour along with oil quenching.

There are various conclusions derived from the studies. These are mentioned as below:

1. The several heat treatments results in the loss of weight. With the soaking time of 2 hours at the temperature of 950°C, the erosion rate is observed to be the minimum in the material subjected to naustenitizing treatment.
2. On comparison with the as received sample, around 41% decrease in erosion rate observed.
3. Distinct treatments of heat has resulted in the changes in the microstructure. Bainite and carbide formation, tempering of martensite were caused due to the treatments of heat generally. Austenite grains coursing is due to the excessive heat input during austenitizing.
4. Several constituents of microstructure affects the behaviour of erosion. Resistance of erosion gets improved with the formation of bainite and tempered martensite. Moreover, particles of fine carbides also result effectively for improving the resistance of erosion. But also, sometimes, the coarse particles of carbides causes deterioration in resistance of erosion.
5. Increase in hardness and UTS results in decrease of erosion resistance.
6. Erosion behaviour of 13/4 martensitic stainless steel gets affected due to the mechanical properties. Resistance of erosion increases with the improvement in toughness and ductility.
7. Silt erosion occurs by ploughing of target surface by hard silt particles. This has been demonstrated by SEM studies. The lip formation is associated with the ploughing leading to the shear. Erosion rates are enhanced as a result of the subsequent removal of lips from the surface of material.

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