# APPRAISAL OF 13 Cr 0.2C STEEL IN AS-ROLLED/HEAT TREATED CONDITION

## A DISSERTATION

submitted in partial fulfilment of the requirements for the award of the degree

of

## MASTER OF ENGINEERING

in

## METALLURGICAL ENGINEERING

(With Specialization in Industrial Metallurgy)

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## CANDIDATE'S DECLARATION

I hereby declare that the work presented in this dissertation entitled "APPRAISAL OF 13Cr 0.2C STEEL IN AS-ROLLED/HEAT TREATED CONDITION" in partial fulfillment of the requirements for the award of the degree of MASTER OF ENGINEERING in Metallurgical Engineering with specialization in *Industrial Metallurgy* of the University of Roorkee, Roorkee, is an authentic record of my own work carried out during the period from July 1996 to March 1997 under the guidance of Dr. A. K. Patwardhan, *Professor & Ex. Head*, *Department of Metallurgical and Materials Engineering*, University of *Roorkee*, *Roorkee*.

The matter presented in this dissertation has not been submitted by me for any other degree or diploma.

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## CERTIFICATE

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

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## ABSTRACT

An experimental study has been made of the effect of heat treatment (hardening followed by tempering) on the mechanical properties of martensitic grade of stainless steel X20Cr13 used in steam turbine applications.

The present investigation was carried out in the form of material/product appraisal. It was observed that hardness in the as quenched condition was a function of the hardening temperature & ranged from 460 to 510  $HV_{30}$ . Tempering lead to a decrease in the as quenched hardness, the magnitude of the decrease being small on varying both, the tempering temperature (660°C & 685°C) & period (90 min to 270 min). Microstructure in the as tempered condition retained the accicularity observed in the quenched condition.

The material behaviour, from the point of view of inclusion rating, was acceptable except for the presence of chunky refractory/slag materials.

Tensile properties in the tempered condition were a function of the quenching temperature as well as tempering temperature & time. The inconsistencies observed in the mechanical properties data could be satisfactory observed/resolved by plotting the mechanical properties as a function of dependent variables and through using the best fit method. Overall the PS ranged from 710-732 MPa, UTS from 776-875 MPa, %EL from 14 to 22 and %RA from 60-68.

Impact toughness improved with tempering temperatures & less clearly with tempering periods. However, at the lower of the two hardening temperatures, the toughness in the as tempered condition showed a more distinctive tendency towards improvement, both with tempering temperature & periods.

Through the above mentioned exercise a correlation is sought to be established between structure and properties. This proved helpful in arriving at understanding of the material's behaviour for the specified application.

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Specimen (Block no.)	Hardening Temperature(°C)	Tempering Temperature(°C)	Tempering Periods (minutes)
1	1010	685	90
2			150
3		1999年1月1日日日日日日日日日日日日日日日日日日日日日日日日日日日日日日日日	210
4		e de la construction de la constru La construction de la construction d	270
5		660	90
6		· .	150
<b>7</b>			210
: 8		n an	270
9	980	685	90
10			150
11		e e e e e e e e e e e e e e e e e e e	1174 - 1922 - 1 <b>210</b> - 1
12			(iii):····: <b>270</b> :
13		660	90
14			1.50 (r. 1.50)
15			210
16			270

## HEAT TREATMENT DETAILS

\* Soaking period for hardening, prior to tempering is 90 min., followed by oil quenching.

\* Tempering treatment is followed by Air Cooling.

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#### **CHAPTER - 1**

## INTRODUCTION

Stainless steels are iron alloys containing a minimum of approximately 11% chromium. The most important among their properties is their corrosion resistance. But for this, they would find little commercial use, as the level of mechanical properties and forming characteristics can be equalled or exceeded by other types of steels at a much lower cost. A chromium content above 12% also provides useful oxidation resistance. Thus the stainless steels are used both for corrosion resisting and high temperature creep resisting heat resisting applications. or Here lies their versatility.

There are a large number of alloys belonging to the stainless steel group and each year new ones/modifications of the existing ones are being added to it. In some steels, chromium content now approaches 30% and many other elements are added to provide specific properties or ease of fabrication, e.g. Ni and Mo are added for corrosion resistance. C, Mo, Ti, Al, Cu for strength, S, Se for machinability and Ni for formability and toughness.

Different grades of stainless steels have been developed for specific end applications. The grade X20Cr13, with which we are dealing in the present work, is particularly useful for steam equipments particularly steam turbine blades, valve bodies, disks and seats and pumps in chemical and petroleum plants. These A400 series stainless steels have a wide range of mechanical properties including high toughness, good resistance to abrasion and to medium corrosive environment.

Looking to the rapid state of industrialization occurring within the country, the power sector is regarded as a basic infrastructure industry. Evidently, the X20Cr13 grade of steel is regarded as a premium grade material of construction for power

generating equipments. Hence a large number of mini steel plants are wanting to/engaged in manufacturing it indigenously, meeting international standards.

The main objective of the present study is (i) to assess whether the experimental material meets with the requirements as laid down in the standard specifications to assess its suitability for particular application area and (ii) to suggest any changes modifications that may be required to be carried out in the manufacturing/processing practices to improve upon the expected level of performance. The various tests performed for the appraisal are instrumental analysis, gas analysis, impact testing, tensile testing, hardenability testing, standard optical metallography, SEM and EPMA etc.

The thesis, which is being presented in the form of an appraisal report, comprises six chapters. The first one outlines the relevance of the thesis, while the second chapter outlines a brief survey of literature on stainless steels in general and X20Cr13 grade in particular. The problem has been formulated in chapter 3, which also contains an outline of the plan of work. The experimental techniques and procedures are discussed in chapter 4. Experimental results are summarized and discussed in chapter 5. Conclusions and suggestions for future work are given in chapter 6.

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## LITERATURE REVIEW

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## 2.1 INTRODUCTION

Stainless steels represent less than 4% of the total amount of steel produced all over the world. However, because they are construction materials for key corrosion resistant equipment in most of the major industries, particularly in the chemical, petroleum, process and the power sector industries, they have a technological and economic importance far greater than could be indicated by the above percentage.

Stainless steels are iron alloys containing a minimum of approximately 11% Cr. This amount of Cr prevents the formation of rust in unpolluted atmospheres [1].

The high corrosion resistance is due to the formation of a thin film of hydrous chromium oxide. The film formed under oxidizing conditions is passive, self healing in character and impervious to further attack. The composition of the film varies from alloy to alloy, and with treatment of the alloy such as rolling, pickling and heating and therefore corrosion resistance also varies [1].

For attaining stainless property in general type of environmental conditions the minimum amount of chromium in the solid solution should be greater than 13% [2]. When Cr is added to steel, it first combines with carbon and forms complex chromium carbides and the remaining chromium goes into solid solution form. Since the amount of chromium combining with carbon is 17 times the amount of carbon [3], the chromium going in the solid solution form will be as follows

Cr in solid solution form = Total Cr -  $17 \times \%$ C. ....(2.1)

Higher the chromium in the solid solution form and lesser the amount of carbides, better is the corrosion resistance.

#### 2.2 COMPOSITION & MECHANICAL PROPERTIES OF STAINLESS STEELS

### 2.2.1 Austenitic Stainless Steels

#### 2.2.1.1 Nickel Stainless Steels

The 300 series represents the largest category of stainless steels produced. Their composition and mechanical properties are shown in Table 2.1 and Table 2.2 respectively. Important compositional modifications in the 300 series to improve corrosion resistance comprise (a) addition of molybdenum to improve pitting and crevice corrosion resistance [4], (b) lowering carbon content or stabilizing with either titanium or columbium plus tantalum to reduce intergranular corrosion in welded materials [4], (c) addition of nickel and chromium in higher amounts to improve high temperature oxidation resistance and strength [4], (d) addition of nickel to improve stress corrosion resistance [4].

Type 304 is the general purpose grade widely used in applications requiring a good combination of corrosion resistance and formability. Type 301 exhibits increased work hardening on deformation and is used for higher strength applications. Type 302 is essentially higher carbon version of type 304, yields higher strength on cold rolling. Type 303 and 303 Se contain sulphur and selenium respectively and are free machining grades used in applications, where ease of machining and good surface finish are important. Type 304L is a lower carbon modification of type 304 used in applications requiring welding. A further modification is type 304N, to which nitrogen is added to enhance strength. Type 305 and 384, which have higher nickel contents, exhibit low work hardening rates and are used in applications where cold formability is important. Type 309, 310, 314 and 330 have higher nickel and chromium contents to provide oxidation resistance and creep strength at elevated temperatures. Types 316 and 317 contain molybdenum and have a greater resistance to pitting in marine and chemical industries. Types 321, 347 and 348 are grades that are used for applications requiring welded structures for elevated temperature service [5].

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## 2.2.1.2 Nickel-Manganese-Nitrogen Stainless Steels

Designated by AISI as the 200 series, their compositions are shown in Table 2.1 and their mechanical properties in Table 2.2. Out of these grades, only types 201 and 202 are produced in significant quantities [5].

Types 201 and 202 have higher yield strengths than their corresponding 300 series counterparts, but their overall corrosion resistance is generally considered inferior to that of the 300 series of steels. A higher molybdenum grade, type 216, is also available that contains upto 0.5 % nitrogen, and is claimed to have good pitting and crevice corrosion resistance [6].

### 2.2.1.3 Higher Alloys

It is convenient to divide these highly alloyed austenitic materials, which contain less than 50% iron, into two groups, namely, Ni-Cr-Fe alloys and Ni-Cr-Fe-(Mo, Cu, Cb) alloys. Typical compositions of Ni-Cr-Fe alloys are given in Table 2.3. Out of these, inconel alloy 600 is used as general purpose corrosion resistant material for elevated temperatures. Inconel alloy 690 is a new material currently being evaluated for steam generation applications [6].

Table 2.4 gives the typical compositions of Ni-Cr-Fe-(Mo, Cu, Cb) alloys. Out of these, Inconel alloy 625 and Hastelloy alloy C-276 are among the most resistant austenitic alloys currently available for severe aqueous environment [7].

2.2.2 Ferritic Stainless Steels

#### **2.2.2.1** The AISI 400 Series

The compositions of the 400 series of AISI grades considered to be ferritic are shown in Table 2.5 and their mechanical properties are listed in Table 2.6. A predominantly ferritic structure should be obtained if the chromium content exceeds approximately 12%, particularly in the presence of other ferrite stabilizers [2].

Recent specifications indicate chromium range to lie between 16 to 18% [6]. The most popular ferritic grade has been type 430, which is the basic 17% chromium stainless steel. Its free machining modifications, types 430F and 430FSe, have also been popular to some extent [5].

In type 405, the addition of approximately 0.2% aluminium, a ferrite stabilizer and maintenance of carbon, an austenite stabilizer, to relatively low levels (about 0.05%) ensures a largely ferritic structure [5]. Types 409 and 439 are stabilized with titanium to improve corrosion resistance at welds in mild environments. Recently, type 409 has been proved useful for automobile emission control equipment. Type 446 is the highest chromium grade in the ferritic AISI 400 series and has the highest corrosion and oxidation resistance of this series [6].

Among the metallurgical problems encountered in ferritic stainless steels are the ductile to brittle transition, 475°C embrittlement, sigma phase formation, high temperature embrittlement, low ductility in the welded condition, and sensitization [2].

### 2.2.2.2 High Chromium Ferritic Stainless Steels

The recent developments in ferritic stainless steels is introduction of high chromium, low interstitial ferritic stainless steels. These developments are linked with new steel making techniques like electron beam melting and large volume vacuum induction melting [8]. The essential feature of these materials is, along with high chromium content, %C + %N should not exceed 0.025%, which keeps the ductile to brittle transition temperature to below room temperature over a wide range of thickness [8]. However, because of the low interstitial levels, extreme care must be taken in welding to avoid the pick up of contaminants from the atmosphere [9]. Laboratory tests have shown that the high chromium plus molybdenum imparts high resistance to pitting and crevice corrosion [10].

### 2.2.3 Martensitic Stainless Steels

#### 2.2.3.1 The AISI 400 Series

These steels contain more than 11.5% chromium and have an austenitic structure at elevated temperatures, that can be transformed into martensite by suitable cooling to room temperature. Thus, martensitic stainless steels by definition lie within the chromium range 11.5-18%, with the lower limit being governed by corrosion resistance and upper limit by the requirement for the alloy to convert fully to austenite on heating [6]. For engineering applications, these 400 series grade steels are used in hardened and tempered condition.

The martensitic stainless steels are generally selected for special applications which in addition to moderate corrosion resistance, require some special combinations of mechanical properties (e.g., high strength with adequate toughness, corrosion plus abrasion resistance, good fatigue resistance after heat treatments, hardenability through thick sections etc.). Their composition and typical mechanical properties are shown in Table 2.7 and 2.8 respectively. These martensitic grade steels are discussed in more detail in the section 2.3 and their applications in section 2.6.

#### 2.2.4 Precipitation Hardening Stainless Steels

The AISI type numbering exists for some of these steels in the 600 series, but these numbers have not been widely used in the technical literature to describe them.

These steels are subdivided into three type as follows.

Martensitic type precipitation hardening steels are generally supplied in the martensitic condition and precipitation hardening is achieved by simple aging treatment of the fabricated part. The precipitation hardening process is thought to involve the formation of very fine intermetallics. Prolonged aging causes these intermetallics, to coarsen, enabling the dislocations, to bypass them during deformation and the strength begins to decline [9]. Generally the aging treatments are designed to optimize high

strength, acceptable ductility, and toughness. Precipitation hardening generally results in a slight reduction of corrosion resistance and an increase in susceptibility to hydrogen embrittlement [9].

The semi austenitic type precipitation hardening steels are supplied in the austenitic condition and this austenite must be transformed to martensite by special heat treatments, before precipitation hardening. This can be achieved in two ways, either by subzero cooling, which is more popular way, or by tempering at about 750°C to reduce the carbon and chromium contents of the austenite by the precipitation of carbides [9].

In the austenitic types, it is the austenite that is precipitation hardened directly. In this type, nickel content is sufficiently high to ensure a fully stable austenite at room temperature. For these steels precipitation hardening is achieved by reheating the austenite to elevated temperatures at which the fine intermetallic compounds are precipitated [9].

#### 2.2.5 Duplex Stainless Steels

Steels containing about 28% chromium and 6% nickel contain both austenite and ferrite and hence are known as duplex. The exact amount of each phase can be varied by the introduction of other austenite and ferrite stabilizers. The compositions of some of the currently available duplex stainless steels are shown in Table 2.9 and their properties in Table 2.10.

The duplex structure renders the propagation of stress corrosion cracks more difficult [4]. Therefore, in the annealed condition duplex stainless steels are regarded as more stress corrosion resistant than some of the lower alloy austenitic grades. The duplex structure is also more resistant to sensitization, but less resistant to crevice corrosion and pitting [10]. The hot working difficulties have retarded the development of duplex stainless steels [4].

#### 2.3 MARTENSITIC STAINLESS STEELS

Martensitic stainless steels can be divided into four groups [11] :

Group No.	Carbon content (%)	Chromium content (%)
Group I	below 0.15	12-14
Group II	0.20-0.40	13-15
Group III	0.60-1.00	14-16
Group IV	0.1	16-18 (Ni 2 to 4%)

#### 2.3.1 Group I [Carbon Content < 0.15%]

The basic physical properties of these steels are recorded as follows [6].

<b>Physical</b>	properties	of	martensitic	steels	with	12-16	%	Cr
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Specific gravity	7.75
Coefficient of expansion (0-100°C)	11 x 10 <sup>-6</sup> per deg C
Thermal conductivity (100°C)	0.06 - 0.07 Cal/cm 5 deg.
Electrical resistivity (20°C)	50-60 micro-ohm-cm
Electrical resistivity (700°C)	110 micro-ohm-cm
Specific Heat (20°C)	0.11 Cal/g deg C
Specific Heat (700 <sup>0</sup> C) Young's Modulus	0.17 Cal/g deg C 12900 tsi

The members of this group are ferro magnetic and remains so whatever heat treatment is applied to them.

The transformation point on heating varies appreciably with the contents of carbon and secondary element such as silicon and nickel; it lies around 900°C, with  $AC_1$  close to 850°C and  $AC_3$  at about 920°C. Thus austenitizing temperatures in excess of 920°C must be used [11]. The inverse transformation on cooling has been investigated under both isothermal and continuous cooling conditions. The results of continuous cooling experiments confirm the extreme stability of the austenite; the critical cooling rate is low and the steel hardens on cooling in air. Maximum hardness only reached after

quenching from 1100°C; the hardness falls off above 1200°C, undoubtedly because a small proportion of  $\delta$  ferrite is present in 13% C steels, it will in fact be seen that the duplex  $\alpha(\delta) + \gamma$  phase field is reached at around 1200°C [11]. Steels of this type are highly sensitive to minor variations in alloy content, particularly with respect to carbon and chromium [2].

### 2.3.2 Group II [ C-0.2 to 0.4 % ; Cr-13 to 15% ]

The physical properties differ very little from those of the Group I steels [6]. It is worthwhile making special mention of one particular physical property of steels in this group, namely their ability to take on a fine polish. Steels can differ one from another in this respect. The differences are associated with the presence or absence of certain non metallic inclusions in the structure [11].

The transformation points on heating are again quite variable. The  $AC_1$  point is around 820°C and the  $AC_3$  point around 860 to 880°C. The austenite is rendered more stable by the higher carbon content. The  $M_s$  point is lowered to as the carbon content is increased, the rapid transformation of austenite is depressed to lower temperatures. No intermediate transformation products are formed. This effect of carbon corresponds to an increased hardening capacity. [11]. The steels will harden by straight cooling in air, even in large sections and are truly self hardening [11]. These steels are normally oil quenched or air-cooled from 1000°C. It should be raised to as high as 1100°C if maximum possible hardness is required [12].

The selection of the quenching temperature, to precede the high-temperature tempering treatment, depends on the result required. To obtain maximum softening combined with maximum toughness, the steel can be quenched from the lowest possible temperature, say 900°C. However if the aim is to obtain optimum toughness at a given hardness value, one may have to resort to higher quenching temperatures [12].

The tempering process and its effect on corrosion resistance have formed the subjects of intensive research, involving in particular the extraction and analysis of carbide precipitates. The carbide  $(CrFe)_3C$  which is formed at the onset of tempering becomes richer in Chromium and eventually transform progressively to  $(CrFe)_7C_3$  first and then to  $(CrFe)_{13}C_6$ . At this stage, the carbide particles start to coalesce. The low corrosion resistance observed at certain stages has been ascribed to impoverishment of the matrix with respect to the chromium in the immediate vicinity of the carbide grains and the consequent formation of local galvanic cells. The effect is said to be at its worst when the carbide present is  $(CrFe)_7C_3$  [11].

#### 2.3.3 Group III [ C-0.6 to 1.0 % ; Cr 14 to 15 % ]

This group, characterized by high carbon contents and an increased chromium content is used when high hardness is essential irrespective of ductility & hardness is the prime consideration, even at some cost of corrosion resistance [6].

In physical properties, these steels differ little from those in group I, apart from a further reduction in thermal conductivity resulting from the simultaneous increases in carbon and chromium contents [6].

The transformation point on heating is lowered by the increased carbon content. Despite this, high temperatures must still be used for hardening, principally with the aim of taking the chromium-rich carbides into solution and ensuring maximum matrix hardness after the quench [11].

Chromium has a significant influence on the as quenched hardness. For steels quenched from  $1000^{\circ}$ C, the hardness falls off substantially as the chromium content is increased from 13 to 17 % [11]. Since a high chromium. content is unavoidable to ensure good corrosion resistance in spite of carbide formation from part of the chromium, a compromise must be made between the two properties; hardness can only be increased at

the expense of corrosion resistance [12]. Provided no significant loss in hardness is involved at the higher tempering temperatures, longer tempering times bring about a useful improvement in ductility [11].

Mechanical properties, as indicated by the tensile and impact tests, are not usually determined on these steels. In fact, they have so little ductility that any results obtained in this way are subject to error and are quite meaningless. Reliance is therefore placed on hardness testing, and selecting a suitable tempering temperature in the range 150-300°C and a time of 1 to 3 hours depending on the relative importance attached to the complementary factors of ductility and hardness.

### 2.3.4 Group IV [ $C \approx 0.1 \%$ ; Cr-16 to 20 % ; Ni-2 to 4% ]

The aim of these steels is to combine the good mechanical properties of the martensitic class with an improvement in corrosion resistance brought about by increasing the chromium content. As the chromium content is increased the steel tends to become 'ferritic' and no longer hardenable. The additional of nickel restores its hardening capacity and transforms it to a 'martensitic' steel [6].

Apart from the possibility of obtaining very favourable mechanical properties by heat treatment, the steel has the further advantage of resisting electrochemical attack in contact with copper alloys, bronzes in particular in the presence of electrolytes such as sea water [11].

The physical properties do not differ notably from those of the other martensitic steels [11].

The transformation points on heating are not known very precisely, and can vary significantly with minor changes in composition. Transformation generally starts at around 650°C and is complete between 800 and 900°C. The micro structure of steel is quite complex. They lie very close to the boundary between the martensite-ferrite and the austenite-martensite-ferrite phase fields. Minor variations in heat treatment

temperature, cooling rates or composition can give rise to major changes in structure. The metal should be mostly martensitic after quenching, but the martensite may also contain islands of  $\delta$ -ferrite, residual austenite in varying amounts or even both constituents together [11].

These group of steels, behave very similarly to the non-stainless-air-hardening constructional steels. Since they have the added advantage of high corrosion resistance and are better in this respect than the 13% Cr steels. They have found many uses in connection with exposure to sea water and saline atmosphere, especially in marine and naval engineering.

Group I steels and the lower carbon members of Group II are used essentially on account of the combination of good mechanical properties and relatively high corrosion resistance. The high carbon members of Group II constitute the cutlery stainless steels, which whilst possessing adequate hardness have a useful degree of ductility.

Group III steels are primarily used for the very high hardnesses they can attain at some cost in ductility. As the carbon content is increased from group to group, the chromium content is also raised to maintain high corrosion resistance despite the possible combination of some of the chromium in carbide form. High chromium content together with a low carbon content greatly improves the corrosion resistance of the group IV steels over that of the previous steels, nickel is added to retain good hardenability and mechanical properties.

#### 2.4 EFFECT OF ALLOYING ELEMENTS ON THE PROPERTIES OF STAINLESS STEELS

Presence of alloying elements help in strengthening one or more of the attributes already stated in Sec 2.2 and Sec 2.3. The effect of prominent elements is discussed below :

## 2.4.1 Effect of Chromium (Cr)

As already stated, Cr provides stainless characteristic. Other alloying elements, however enhance the effect of Cr in many environments and impart many of the special properties [6].

Chromium. has been observed to increase resistance to tempering by decreasing the kinetics of the precipitation and growth of carbides in the matrix. Accordingly it is expected that the presence of stronger carbide formers will prove more effective in this regard.[4]

Chromium. is reported to increase the wear resistance of martensitic stainless steels. In addition a slight beneficial effect in ferritic and austenitic stainless steels can be expected due to solid solution hardening [13]. chromium is thought to have no effect on fatigue strength of steel [13]. Reportedly additions of chromium have little effect on creep strength [13]. In general; the ferritic stainless steels have low impact strength and are relatively notch-sensitive at ambient temperatures, especially when chromium is about 20 to 21% [4]. Hardness & tensile strength increases, with increasing chromium content. [12]

The increased resistance to tempering imparted by chromium. acts to enhance the elevated temperature strength of the martensitic stainless steels [4]. In the non hardenable grades, however chromium contributes little to strength at the higher elevated temperature. In high temperature service the primary effect of chromium is to maintain the integrity of the steel by imparting resistance to oxidation [4].

The increase in hardenability due to addition of chromium, to carbon steel increases the susceptibility of hydrogen induced cold cracking in the heat affected zone (HAZ) of martensitic stainless steels [9].

#### 2.4.2. Effect of Nickel (Ni)

Nickel is the most important metal deliberately added to high chromium. steels to improve properties. The addition of Ni restores the ability of the steel to transform to martensite on quenching or air cooling due to its being a  $\gamma$  stabilizer. This gives beneficial effect on the mechanical properties of steels.

Nickel is generally added to martensitic stainless steel castings to improve strength, ductility and corrosion resistance. For example, Souresny and Saver developed a nickel containing 13% Cr cast steel for highly stressed components of hydraulic power plants. They found that increasing Ni from 1.25 to 4% improves cavitation resistance, sand erosion resistance and alternating bend fatique strength in the wet and dry state when tested using notched and smooth bar specimens [4].

#### 2.4.3 Effect of Molybdenum (Mo)

The principal function of Mo as alloying element in stainless steels are :-

- (i) Improvement of the corrosion resistance of austenitic and ferritic stainless steels.
- (ii) Improvement of the elevated temperature mechanical properties of austenitic stainless steels.
- (iii)Improvement of the strength and resistance of tempering of martensitic stainless steels.

The martensitic stainless are employed in application requiring a combination of high strength plus a limited degree of corrosion resistance. Molybdenum additions (0.5 to 4%) increase the tempering resistance and intensify the secondary hardening reaction of the 12% Cr martensitic stainless steels. Mo also improves room temperature, Ultimate Tensile Strength and Yield Strength and resistance to deformation at elevated temperatures [2].

The martensitic stainless steels are widely used in steam power plants. For such components such as turbine blades. Molybdenum additions increases the maximum permissible operating temperatures for such parts [2].

#### 2.4.4. Effect of Vanadium (V)

Vanadium improves the pitting resistance of stainless steels particularly in the concentration range of 2.5% [4]. An additional advantage of increasing amount of Vanadium in ferritic stainless steels is the improvement it confers on the impact properties an aspect in which these steels are very deficient [4]. The combination of V with Mo also had a beneficial effect on the pitting potential in the cast austenitic alloys [8].

The effect of vanadium in the cast austenitic alloys was to increase the critical current density and to contract the potential range of the passive region and to move it to higher current values [8].

An important difference between Mo and V as alloying elements is the Mo occurs largely in solid solution where as Vanadium occurs both in solid solution and as a precipitate [4].

## 2.5 FACTORS IN SELECTION OF STAINLESS STEELS

The first and most important step in the selection process is the selection appropriate to the application. A number of standard grades exists that differ from one another in composition, corrosion resistance, physical properties and mechanical properties. Selection of the particular grade with optimization of properties for a specific application is the key to satisfactory performance at a minimum total cost.

A checklist of characteristics to be considered is given below [9]. The important characteristics are discussed in brief.

- Corrosion resistance
- Resistance to oxidation and sulphidation
- Strength and ductility at ambient and service temperatures.
- Suitability for intended fabrication techniques.
- Stability of properties in services.
- Toughness
- Resistance to abrasion and erosion.
- Surface finish obtainable
- Magnetic properties
- Thermal Conductivity
- Electrical conductivity

#### 2.5.1 Corrosion Resistance

It is the most important characteristics of a stainless steel, but often is also most difficult to assess for a specific application. General corrosion resistance to natural conditions and to pure chemical solutions is comparatively easy to determine and is determine by the standard weight loss method [7].

General corrosion is often much less serious than localized forms such as stress corrosion cracking, crevice corrosion in tight spaces or under deposits, pitting attack and intergranular attack in sensitized material such as various heat affected zones (HAZ) in fabricated parts. Such localized corrosion can cause unexpected amd sometimes catastrophic failure, while most of the structure remains unaffected and therefore must be considered carefully in design and selection of the proper grade of stainless steel.

Corrosive media may differ substantially due to slight variation in some of the corrosion factors listed below [9].

- Chemical composition of the corrosion medium including impurities.
- Physical state of the medium-liquid, gaseous, solid or combinations thereof.

#### - Temperature variation

- Oxygen content of the medium

- Continuity of exposure of the metal to the medium etc.

Even slight variation in one of the above factor, may lead to substantial variation in the performance of the material for particular application and i.e. why corrosion resistance is considered to the most difficult property to assess for particular application.

#### **2.5.2** Mechanical Properties

Mechanical properties at service temperature obviously are important, but satisfactory performance at other temperatures must be considered also. Thus a product for arctic service must have suitable properties at subzero temperature even though steady state temperatures may be much higher. Room temperature properties after extended service at elevated temperature can be important for applications such as boiler and jet engines, which are intermittently shut down [13].

#### 2.6 APPLICATION AREAS OF STAINLESS STEEL

Application of stainless steels in general and martensitic grade stainless steels in particular are discussed below [4].

#### Application in marine systems [4]

Austenitic and martensitic stainless steels have been used in marine power plants as super heater tubing and turbine blading respectively. Most grades of stainless steel perform satisfactory in the marine atmosphere, except that certain grades discussed below may be susceptible to stress corrosion cracking. The martensitic steels, type 410 may rust on few months in the marine atmosphere. General rusting of the stainless steels in seawater is of little or no practical concern. Functional application in marine system involve, propellers (CF-8, A410), pumps (CF-8M, AISI 316), bulk tankages (AISI 304L and 316L), heat exchangers (AISI 316), O ring seals (AISI 304 and AISI 316).

#### Application in the chemical and process industry [4]

All of the various grades of stainless steels are used at various places in the chemical industry. However, the major tonnages are in the austenitic grades (type AISI 304, 304L, 316 & 316L) the ferritic grades of type 430 and for casting ACI grades CF-8, CF-8M-CN-7M.

Type A430 is widely used for all types of equipment in the ammonia oxidation process for nitric acid manufacture and may other components in nitric acid medium. Type A403 grades were employed for a long time in industry but now it has been largely replaced by less costly cast austenitic grades.

### Application in transportation system [4]

The 11% Cr or type A409 is being used increasingly to replace M.S. in specialized applications. One such application is for self contained tanks for fire fighting apparatus. Because such equipments must be ready for used at any instant, the storage tanks are always filled with water. A409 grade steel provides adequate corrosion resistance and is fabricated readily. Other applications includes the joints used in exhaust system i.e. where temperature and corrosion condition are modest which allows use of type A410 and A409 grade steels.

### Applications in the food Industry [4]

Various grades of stainless steels are being widely used in the food industry for its cleanability. Hardenable grades such as type A420, A409 through A316 are used in plows and planters as they provides long measurably corrosion and abrasion resistance in adverse conditions, as soil may prove very abrasive and, at time corrosive.

### Applications in petroleum industry [15] :

In prevention of hydrogen sulfide attack, 400 series stainless steels and precipitation hardenable stainless steels are used.

The 12% Cr stainless steel, specifically types A410, A410S and A405, find applications as cladding, exchanger tubing, ballast trays, pump components etc. In recent years type 430 has found significant usage as welded heat exchanger tubing, replacing lower chromium alloys because of lower cost and superior corrosion resistance.

### 2.7 PERFORMANCE OF THE MATERIALS USED FOR TURBINE BLADES.

Heat resistant, low alloy, chromium containing structural steels, containing less than 12% Cr and ferritic stainless steels containing more than 12% Cr, are the materials potentially used in steam generation plants. The temperature limits in conventional steam power stations is restricted to about 813K to 823K or to tube wall temperatures upto 843K (564°C). Above that temperature modified chromium steels (app. 12% Cr) are required. At temperature from about 863K and over, only austenitic chromium nickel steels or nickel base alloys are eligible [14].

#### 2.7.1 Probable causes of failure [15] :

Severe damage to turbine blades of material, U.S. standard X15Cr13 was observed as a result of pitting and blade rupture in the zone of the one rotar series of a condensation turbine. The damage occurred after about 5 operating years was caused by chlorides from insufficiently demineralised feed water.

Stress corrosion cracking (SCC) of low alloy steels and the crack formation in high purity steam is ascribed on the basis of recent studies, to non metallic inclusion in the steel which act as a starting site for pitting, the resulting pits becoming starting sites for crack initiation. Apart from chlorides,  $SiO_2$  deposits can also cause severe turbine damage, complete write-off damage occurred after eighteen months operation on rotor blades made of steel X20CrMo13 (Cr-12 to 14 % & Mo- 1 to 1.3 %) owing to inadequate silicate removal in the feed water treatment.

Erosion is relatively frequent in the high pressure section of saturated steam turbines, within the range of high steam densities, in external water separators, super heaters and in the final turbine stages. The erosion risk is reduced by using 13% Cr steel for complete blading. Moreover, treatment of critical parts by superheated steam prior to starting operation has been recommended as a remedy against erosive damage. This treatment produces a homogeneous protective magnitite layer which is resistant to erosion. A further measure aimed at preventing erosive damage in wet steam turbines, particularly on the turbine blades, is the use of film forming amines, for e.g. octadecylamine in combination with a volatile steam alkalinizing agent preferably morpholine.

The collective experience gained from years and years together on the performance of materials used for turbine blades, a few standard grades are recommended for the use. These are as follows [15].

1. U.S. standard X7CrAl13 (DIN Mat. No. 1.4002)

2. U.S. standard X8Cr13 (DIN Mat. No. 1.4016)

3. U.S. standard X8Cr(Ti)Nb 17 (DIN Mat. No. 1.4511)

4. U.S. standard X15Cr13 (DIN Mat. No. 1.4024)

5. U.S. standard X20Cr13 (DIN Mat. No. 1.4021)

6. U.S. standard X22Cr Mo V121 (DIN Mat. No. 1.4136)

7. U.S. standard X40Cr13 (DIN Mat. No. 1.4034)

8. U.S. standard X110Cr Mo 17 (DIN Mat. No. 1.4126)

However, and argument against the use of heat treatable and martensitic grade, chromium steels in boiling water reactors is their suspectibility to Stress Corrosio Cracking (SCC) in steam. A further point is the susceptibility to inter crystaline corrosion following welding.

#### 2.8 CONCLUDING REMARKS

In this chapter, after a brief introduction to stainless steels in Sec 2.1, are classified and described in the Sec 2.2. Martensitic grade stainless steels are discussed in more details in the Sec.2.3. Sec. 2.4 comprises the effect of alloying elements (Cr, Ni, Mo, V) on the properties of the steels Sec. 2.5 outlines the factors used for selection of stainless steels, while Sec. 2.6 discusses the application area of stainless steels and martensitic grade stainless steels in particular. The last i.e. sec. 2.7 outlines the observations made on the performance of the materials used for turbine blading. The abovementioned information is useful in formulating the problem.

#### CHAPTER - 3

## FORMULATION OF THE PROBLEM

#### **3.1 GENERAL**

This chapter comprises an interplay between the basis of materials selection and the need for product/materials appraisal culminating into the formulation of the problem.

#### **3.2 MATERIALS SELECTION**

Factor affecting materials selection are, corrosion resistance, resistance to oxidation and sulphidation, suitability for intended fabrication technique, stability of properties in service, adequate mechanical properties and surface finish obtainable etc. [9].

In principle, one could write a mathematical expression describing the merit of an engineering design as a function of all these variables, differentiate it with respect to each of the criteria for evaluation and solve the resulting differential equation to obtain an ideal solution. In practice, while dealing with real life problems such an easy method is not possible. In some instances, a standard readily available component may be much less costlier than, and yet as effective as an optimized component.

In brief material selection is carried out in three steps. First, the designer must make a systematic study of the intended purpose of the product under design. The principal result of this study is characterization of the functional requirements of the product, including the relative priorities of potentially conflicting requirements.

In the second step, these characteristics should not be considered merely as numbers in published data related to properties. They are better described as the

response of the material in the form of specific component to the imposed service conditions. The effects of manufacturing processes on material properties are implicitly considered when the material is evaluated as a specific component.

The third step in the design process is to consider various combination of configuration material and fabrication process, evaluating each against previously established criteria and making changes accordingly [16].

### 3.3 IMPORTANCE OF THE PRODUCT APPRAISAL

Two mostly found reasons for selecting a certain material for a particular application are

a) the material has always has been used in that application and

b) the material has the right properties.

Neither is evidence of original thinking or even careful analysis of the application. The time has passed when each application had its preferred material and a particular material is having it's secured market. In this context, the term property is something that a material inherently possesses. On the contrary, a property should be regarded as the response of the material to a given set of imposed conditions. It also be recognized that this property should be that of the material in its final available and processed form [16].

The collective usage of a material in a particular application is useful information, but not justification in itself for selecting a material. Tabulated properties data, such as those available from the sources like American Society for Metals, American Iron and Steel Institute, American Society for Testing and Materials, Society of Automotive Engineers, API standards etc., is helpful, but such information must be used judiciously and must be relevant to a particular application [9].

#### **3.4 WORK PLAN**

#### 3.4.1. Objective

Having thus laid down the modalities of materials selection & it's inter relation with product appraisal, it is necessary to formulate the problem. The review of literature has highlighted the importance of the higher chromium steels in hightech applications.

The main objective of this product appraisal was to assess whether the experimental material i.e. X20Cr13 which is in a developing stage in some of the mini steel plants, is meeting the specifications as described in the standards i.e.namely AISI 420 (DIN 17440) [17]. Appraisal has been carried out mainly in the heat treated condition and related to a particular application area i.e. steam turbine blades.

#### 3.4.2 Phase Wise Plan

The phase wise planning relating to the investigation has been given below : **PHASE-I**.

Validation of chemical composition and effect of heat treatment on the hardness, microstructure and grain size along with inclusion count, in the hardened & tempered condition.

#### PHASE-II

Effect of heat treatment on the mechanical properties like U.T.S., Y.S., % Elongation, % reduction in area and impact strength.

#### PHASE-III

Fractographic study of selected impact and tensile tested specimens ; other miscellaneous studies such as EPMA for analysing the nature of inclusions in quantitative manner and gas analysis.

#### **CHAPTER - 4**

## EXPERIMENTAL TECHNIQUES AND PROCEDURES

#### 4.1 MATERIAL

The material used for this study was 13 chromium martensitic stainless steel conforming to the specification AISI 420, DIN 17440 [17]. It was made available by M/s Star Wire (India) Limited, Ballabhgarh in the form of slabs, size ( $40 \times 22 \times 1100 \text{ mm}$ ). The allowable compositional variation for this grade of steel is as shown in the Table given below :

Alloying elements	Cr	Ni	C	Mn	Si	S	Р
%	12.5-14	0.3-0.8	0.17-0.22	0.3-0.8	0.1-0.5		0.03 max

#### **4.2 HEAT TREATMENT**

Mainly two types of heat treatment were carried out comprising hardening and tempering. They were carried out in muffle furnaces whose temperature was controlled to  $\pm 5^{\circ}$ C.

The temperatures suggested in the standards are  $980^{\circ}$ C to  $1030^{\circ}$ C for hardening and  $>650^{\circ}$ C for tempering. Hardening was carried out from  $980^{\circ}$ C and  $1010^{\circ}$ C after soaking for one and half hour and followed by oil quenching. Tempering was carried out at  $685^{\circ}$ C and  $660^{\circ}$ C for 1.5, 2.5, 3.5, 4.5 hours respectively, followed by air cooling. Thus a total of 16 heat treatments were carried out i.e. for each hardening temperature, two tempering temperatures and for each tempering temperature, four holding periods were used.

#### **4.3 PREPARATION OF TEST SPECIMENS**

From 16 heat treated blocks of size (40 x 22 x 60), 32 round tensile test specimens, 16 Charpy impact specimens and 16 specimens for optical metallography (10 x 20 x 12 mm) were cut, i.e. 2 tensile test specimens, 1 charpy impact test speciment specimen and one speciment for optical metallography were obtained from each heat treated block.

Round tensile specimens were used to determine the effect of heat treatment on the tensile behaviour. Fig. 4.1 gives details of specimens.

Charpy V-notch impact test was used to measure the fracture toughness in terms of Charpy, V-notch energy. The specimen details are given in Fig. 4.2 and are as per specification AWS B70-77 [18].

Optical microscopy was extensively employed to assess the effect of heat treatment on the microstructure, grain size and for the study of inclusions present in the material. The specimens were polished, adopting standard technique/procedure, upto 4/0 grade of emery paper and then cloth polished.

The specimens used for evaluating the effect of heat treatment on the hardness were cut from the heat treated blocks and polished, using standard techniques, upto 2/0 grade of emery paper.

#### 4.4 INSTRUMENTAL ANALYSIS

Instrumental analysis was carried out, for validation of chemical composition, on the vacuum quantometer from B.H.E.L., HEEP, Hardwar.

#### 4.5 METALLOGRAPHIC STUDIES

Optical microscope MeF-3 was employed for the metallographic studies. Microphotographs were taken at 2 magnifications, 100X and 400X in unetched condition on selected specimens for detailed study of inclusions and their rating. For the study of microstructure and grain size, specimens were etched with freshly prepared etchant, ferric chloride of the composition (FeCl<sub>3</sub>-5 gm + HCl-50 ml, H<sub>2</sub>O-100 ml + 2 to 3 drops of HNO<sub>3</sub>) [19]. Subsequently and then microphotographs were taken at two magnifications, 100X and 400X for grain size and microstructure respectively.

#### 4.6 MECHANICAL TESTING

Mechanical testing involved assessment of hardness, impact and tensile properties in the heat treated conditions. Test specimens were made from longitudinal orientation only.

#### Hardness Measurements

Hardness measurement was done on Vickers hardness testing machine using a load of 30 kg. A minimum of eight impressions were taken on each specimen at various locations, and average of these values, along with standard deviation recorded.

Impact Testing

Impact tests were conducted at room temperature on standard Charpy V notch specimens as per ASTM E-23 standard [20]. Pendulum type impact testing machine with a capacity of upto 2 tons was used to carry out the impact test.

#### **Tensile Properties**

Tensile tests were conducted on cylindrical samples made as per the details shown in fig. 4.1, on an Instron testing machine, model 1342 at two strain rates , 1mm/min and 50 mm/mim respectively and values of the Ultimate Tensile Strength (UTS), Proof Strength (PS), Elongation (%EL) and % Reduction in Area (%RA) were calculated and recorded.

#### **4.7 FRACTOGRAPHIC STUDIES**

Fractographic studies were conducted on selected tensile and impact te specimens. These studies are necessary to evaluate the nature of fracture and relation to the manufacture and properties. Subsquently microphotography were take various magnifications. Phillips Scanning Electron Microscope (SEM-501) operating an accelerating voltage of 15 kV was employed for this purpose.

#### **4.8 EPMA**

Electron probe Micro Analysis was carried out on the JEOL, JXA-8600M, Elec Probe Analyser. These studies are helpful in finding out the trace elements in inclusions. One specimen, prepared for Metallographic study was ued in Electron Pr Micro Analysis.

#### 4.9 GAS ANALYSIS

Gas Analysis was carried out for  $O_2$  and  $N_2$  from B.H.E.L., HEEP, Hardwar. <sup>r</sup> speciments were used for each analysis to check the reliability of the results.

#### CHAPTER - 5

# **RESULTS AND DISCUSSIONS**

#### 5.1 GENERAL

In this chapter, results/data relating to instrumental analysis and effect of heat treatment on the hardness, microstructure and grain size, alongwith inclusion count are reported and subsequently discussed. The effect of heat treatment on the tensile properties and impact toughness is reported mostly in tabular and graphical forms. Mechanical properties thus obtained are co-related with the microstructure in the subsequent discussion. Finally, gas content ( $O_2$  and  $N_2$ ) is reported. An overview in the form of product appraisal is then put forward.

#### 5.2 RESULTS

#### **5.2.1** Chemical Analysis

Instrumental analysis of the experimental material is reported in Table 5.1. It was observed that the analysis conforms with that as specified in the AISI 420 [17].

#### 5.2.2 Effect of Heat Treatment on Hardness & Microstructure

Data obtained in this regard is summarised in Table 5.2 and Figures 5.1 to 5.5. A perusal of the relevant figures and table revealed as follows :

- 1. The as quenched hardness was a function of the hardening temperature, it being higher at higher temperature.
- 2. The as quenched hardness was lower than what is normally attained in higher carbon steels and that attained in low carbon, medium alloyed and highly alloyed steels.

- 3. Tempering lead to a decrease in the as quenched hardness at both the tempering temperatures, 685°C and 660°C. The hardness decreased marginally with an increase in tempering period.
- 4. The above mentioned changes in hardness were consistent with the microstructure observed (Figs. 5.3 to 5.5).
- 5. The microstructure in the as quenched condition was accicular in nature. That it has been formed by shear can be adjudged on the basis of surface relief (Fig. 5.3).
- 6. Tempering temperature and soaking period had little effect on the accicular nature of the as quenched microstructure (Figs. 5.4 and 5.5 A-D).
- On increasing the soaking period at given tempering temperature, the only discerning change was perhaps a slight coarsening of the microstructure (Figs. 5.4 and 5.5).
- Representative high magnification microstructures (Figs. 5.4 E & F, Figs. 5.5 E & F) proved helpful in ascertaining that the basic nature of the as quenched accicular microstructure had remained more or less unaltered.

#### **5.2.3 Nature of Inclusions**

Representative microphotographs in this regard are summarized in Fig. 5.6. It was revealed that, inclusions mostly comprised oxides (Fig. 5.6 A). Occasionally, MnS stringers were also present. They appeared as light grey, thin thread like features (Fig. 5.6 B)[21]. The nature of certain bead like features seen in (Fig. 5.6 C) could not be ascertained [22]. The chunky inclusions shown in Fig 5.6 D comprised entrapped refractory/slag materials [21].

Electron probe micro analysis of randomly selected regions delineating the type of inclusions shown in Fig 5.6 A & B revealed that they comprised mostly FeO and  $Cr_2O_3$  (Table 5.3-A & B). Where as in one of the instances, presence of  $K_2O$ ,  $Na_2O_3$ , MnO and

CaO was revealed (Table 5.3-A), in the other instance the inclusions mostly comprised  ${}^{c}$ FeO and Cr<sub>2</sub>O<sub>3</sub> along with SiO<sub>2</sub> and MnO (Table 5.3-B).

#### 5.2.4 Effect of Tempering on the Tensile Behaviour

The effect of tempering temperature and time on the tensile behaviour is summarized in table 5.4, and in the Fig 5.7 to 5.14. A perusal of the data contained in table 5.4 revealed that an increase in tempering period at a given tempering temperature had little effect on the UTS & 0.2% PS. If any, there was at best a marginal decrease in both of these parameters. Any improvement in % RA & % EL was not much in evidence.

Quenching temperature had an effect on the mechanical properties in the as tempered condition, for e.g. on hardening from temperature 1010°C, the UTS & PS values in the as tempered condition, overall, were higher than those obtained on hardening from 980°C. However, as had been observed earlier, the corresponding variation in % RA & % EL values didn't reveal a consistent trend.

It further emerged that the % EL & % RA values improved with tempering period, on tempering at lower of the two tempering temperatures. This observation was valid for both the quenching temperatures.

The effect of increasing the strain rate by a factor of 50, on the tensile properties didn't reveal a consistent trend. Whereas in some instances, an increase in the UTS & PS values was observed, in most other situations, a small decrease in both these parameters was observed. A similar situation also existed for % EL & % RA values.

The aforementioned changes in properties, brought about by varying the tempering parameters, have been graphically represented in the Fig 5.7 to 5.14. The basic purpose behind such an exercise was to assess in what way the experimentally observed variations differed in comparison to the expected/idealized ones. This aspect has been critically analysed in Sec. 5.3.

#### 5.2.5 Effect of Heat Treatment on Impact Toughness

The data pertaining to this aspect has been summarized in the table 5.5 and in the Figs. 5.15 & 5.16. An analysis of the data contained in the table 5.5 revealed that :

1. For the 1010°C OQ + tempered state,

- (i) The toughness values on tempering at 685°C, (average around 33 J) were better than those attained on tempering at 660°C (average around 30 J).
- (ii) On tempering at 685°C and on excluding the toughness attained on tempering at three and half hours, the other values are more or less comparable.
- (iii)A similar situation was observed to exist on tempering at 660°C, after excluding the toughness value for one and half hour.

2. For the 980°C OQ + tempered state,

- (i) On tempering at 685°C, a continuous improvement in toughness with tempering period was observed.
- (ii) On tempering at 660°C, inconsistencies as observed in the 1010°C OQ + tempered condition were once again in evidence.

3. On comparing the toughness values as influenced by the quenching temp., it emerged that the specimens hardened from 980°C attained better overall toughness than those hardened from 1010°C. This observation was valid for both the tempering temperatures.

Plotting the impact toughness data as a function of hardening/tempering temperatures as influenced by tempering period, proved helpful in rationalising the impact behaviour of the experimental material. This aspect has been discussed in detail in the Sec. 5.3.

#### 5.2.6 Fractographic Studies

Such studies were carried out on selected impact tested as well as tensile tested specimens. Representative fractographs are summarized in the figs 5.17 & 5.18, (impact tested) and figs. 5.19 & 5.20 (tensile tested).

Considering first the fractographic observations on impact tested specimens, it emerged that

- (i) The fracture appearance was brittle within the toughness range 23 to 35 J(Fig 5.17 A to D, Fig 5.18 A).
- (ii) A careful perusal of these fractographs revealed the existence not only of transgranular brittle fracture, but more importantly of faceting indicative of intergranular brittle fracture (Fig. 5.17-A to D, Fig. 5.18-A&B).
- (iii)On approaching an impact toughness value of around 42J, the fracture appearance, although still brittle (Fig. 5.18-B), revealed features indicative of gross deformation.
- (iv) The fracture appearance corresponding to the highest toughness value revealed the existence of gross deformation in the form of shallow ductile dimples (Fig. 5.18-C). Additionally the existence of secondary cracking was till in evidence.
- (v) Gross deformation was observed to persist even at a toughness level of around 44J (Figs. 5.18-D).

Commenting now on the fractographs of tensile tested specimens, it emerged that although considerable micro cracking was observed (Fig.5.19 & 5.20A to D), most of the fractographs did reveal the existence of gross deformation in varying amounts. The extent of gross deformation was found to be a function of the ductility/toughness of the material, for e.g. specimen corresponding to Fig. 5.19-B, exhibiting toughness higher than the other specimens (Fig. 5.19 A,C,D), revealed a comparatively large degree of gross deformation. Similarly amongst the fractographs, summarised in Fig.

5.20, the one at Fig. 5.20-B revealing gross deformation higher than the others, exhibited a toughness of 52J. Thus in a way, the fractography carried out on tensile tested specimens gave a good account of ductility/toughness of the experimental material.

#### 5.2.7 Gas Analysis

The results of gas analysis carried out are summarised in Table 5.6. From the data, it emerged that, whereas the oxygen levels are within the acceptable limits, the nitrogen content was found to be substantially higher [23].

#### 5.3 DISCUSSIONS

The results outlined in Sec. 5.2 can be appropriately discussed in the light of the structural changes that take place during hardening & tempering treatments. The starting point for such an analysis is the composition of the material. The composition summarised in Table 5.1 is consistent with that stipulated in relevant specifications. This provides reaffirmation that the experimental material is a martensitic grade of stainless steel and hence will respond to the hardening and tempering treatment.

The hardening and tempering temperatures used are as provided in the specifications. The range of hardening temperatures is stipulated to be 980°C to 1030°C and the preferred tempering temperatures are  $\geq 650^{\circ}$ C [17]. The second hardening temperature was chosen as 1010°C to avoid grain coarsening. Similarly, the tempering temperatures selected are 660°C and 685°C. While deciding these temperatures, due consideration was paid to the thumb rule that growth rate doubles with every 50°C rise in the heat treating/processing temperatures.

Hardness in the as quenched condition, although lower than what is attained in higher carbon and higher alloyed steels, is consistent with the carbon content [4]. The presence of chromium has ensured that the material has the requisite hardenability to

transform to accicular/shear tranformation structure (Table 5.2 and Fig. 5.1). Higher the hardening temperature, higher will be the hardness due to an increase in hardenability. This is what has been observed.

Tempering from 685°C and 660°C has lead to a decrease in hardness due to partial decomposition of the hardened accicular structure through the expected carbide transformation sequence, namely :

Shear structure  $\rightarrow$  Fe<sub>2.4</sub>C  $\rightarrow$  Fe<sub>3</sub>C  $\rightarrow$  (FeCr)<sub>x</sub>C<sub>y</sub>  $\rightarrow$  Coarsened (FeCr)<sub>x</sub>C<sub>y</sub> or its variants

Soaking period had a very small effect on as tempered hardness, because the overall level of hardness has apparently reached the 'level off' stage (Table 5.2 and Figs. 5.1 and 5.2) and also because the soaking temperatures/periods used are not large enough to affect substantial changes in microstructure/hardness. Besides the carbide transformation, the other factor that promotes retention of a higher hardness is the stability of accicular structure even in the as tempered condition (i.e. it is not transforming to a combination of polygonal ferrite and dispersed carbides). This analysis explains the reasons for a similarity between the as quenched and as tempered microstructures (Figs. 5.3 to 5.5).

The presence of surface relief in the as quenched condition is a confirmation that the as quenched microstructure is formed by shear (Fig. 5.3). The slight discerneable coarsening on increasing the tempering period is due to initiation of the tendency of accicular structure to transfom into a polygonal structure. Representative high magnification microphotographs (Figs. 5.4 and 5.5 - E and F) have been included, just to show that the structure obtained under the existing tempering conditions is not polygonal in nature.

Considering now the nature of inclusions, (Figs. 5.6 A to D, section 5.2.3), the inclusion rating for the experimental material comprised;

(i) Oxide type - Heavy Series - C

(ii) Sulphide type - Thin Series - B

Thus, the overall inclusion distribution in the experimental material may be regarded as satisfactory.

Electron Probe Micro Analysis of randomly selected regions gave a further insight into the constitution of the oxide inclusions (Table 5.3 A & B). Interestingly, the constitution of the oxides in the two situations differ significantly. It would be worth examining whether this is a satisfactory situation from steel making point of view or not, especially so, since the cracks are known to originate from regions containing non metallic inclusions [15]. There are instances to suggest that such cracks can eventually lead to premature failure [15]. That being so, inclusion's shape, size, nature and distribution become important parameters in counteracting such tendencies. In this regard the entrapment of large sized refractory/slag materials (Fig. 5.6 D) is not a desirable feature.

Having thus far discussed the changes in hardness and microstructure, it would now be pertinant to consider the effect of tempering parameters on the tensile properties. A marginal decrease in UTS and PS on increasing the soaking period, at a given tempering temperature would be on account of slight coarsening of dispersed second phase particles. No evidence can be provided to this effect as TEM studies have not been carried out. However, microstructural studies have indicated that the accicularity of the hardened structure is maintained even in the tempered state. As no other structural change is occuring, it would not be incorrect to infer that the marginal decrease in tensile properties could be on account of particle coarsening. Perhaps the decrease in UTS or PS is small enough, not to have an appreciable effect on either % EL or % RA, as the experimental data suggests (Table 5.4).

Quenching from a higher temperature, i.e. 1010°C has lead to the attainment of higher UTS and PS values than those obtained on hardening from 980°C primarily because a higher hardness was obtained on hardening from 1010°C. The absence of a consistent

trend in % EL and % RA values could be on account of one of the following reasons :

(i) The microstructure being non homogeneous due to particle coarsening.

(ii) Lesser number of samples having been used per test performed.

The % EL and % RA values improved with tempering period at the lower of the two tempering temperatures, since tempering at 660°C ensured a greater uniformity in the microstructure vis a vis dispersed carbide particle size, shape and distribution. Such a microstructure is more useful in regulating the deformation, leading to the attainment of better ductility.

In order to arrive at a rationalised behaviour of the experimental material in the quenched and tempered condition, the mechanical properties, summarised in table 5.4, have been graphically represented in figs. 5.7 to 5.14, as a function of hardening temperature and tempering variables. Both, the actual variations and the best fit variations have been indicated. As mentioned in Sec. 5.2.4, such representations are useful as they reveal the deviations from the idealised/expected behaviour. The best fit plots, in fact are useful in projecting the likely/expected mechanical properties for a given heat treating/processing conditions. The likely reasons for the deviations from idealised/expected behaviour are as follows :

- (i) The material composition being not uniform which is often the case under practical heat treating conditions.
- (ii) The possibility of there being improper temperature controls while heat treating.

(iii)Lesser number of samples having been used per test performed.

Before concluding the discussion on the effect of tempering parameters on the tensile properties, it would be of interest to mention that the propeties obtained in the present study are comparable with those indicated in the standard specifications [17].

The impact toughness values summarised in table 5.5 and in figs. 5.15 and 5.16,

provide useful pointers to the overall suitability of the material for the applications specified. The overall toughness in the as tempered condition on quenching from 980°C is better than the one obtained on quenching from 1010°C due to a favourable austenite grain size. Its usefulness is further reflected even on tempering from both the temperatures.

The toughness values on tempering at 685°C are better than those obtained on tempering at 660°C because of a slightly lower, overall strength obtained in case of the former.

Looking at the toughness values in the tempered condition, obtained on hardening from  $1010^{\circ}$ C, it is seen that, although the toughness on tempering at  $685^{\circ}$ C is marginally better (33J) as against that obtained on tempering at  $660^{\circ}$ C (30J), the overall improvement in the toughnes with an increase in tempering period is rather small, as revealed through the best fit plots (Fig. 5.15). Clearly the likely improvement has been offset by the presence of a relatively coarser austenite grain size and a possible non uniformity in the size, shape and distribution of dispersed carbide particles.

The data summarised in fig. 5.16 obtained on hardening at 980<sup>o</sup>C followed by tempering duly confirms the above reasoning to be correct, e.g. the best fit data line which is nearly horizontal for both the hardening temperature is more favourably disposed at the lower of the two hardening temperatures (Figs. 5.15 & 5.16); similarly the toughness values on tempering at 685<sup>o</sup>C show a continous increase in toughness (Fig. 5.16), unlike what is observed in fig. 5.15, wherein the toughness is remaining more or less unchanged with tempering period. Thus preparing the best fit plots has once again proved useful in rationalising the impact behaviour of the experimental material.

The overall toughness values obtained in the present study are comparable with those specified in the standards [17].

Considering now the inferences drawn on the basis of fractographic studies, the

toughness values in the range of 23J to 35J are definitely low and expectedly have induced the material to fracture in a brittle manner (Fig. 5.17 A to D & Fig. 5.18-A). Generally transgranular quasi-cleavage fracture is expected. However the existence of faceting of intergranular brittle fracture (Fig. 5.17A to D, Fig.5.11 A&B), is somewhat of an unexpected feature and may be attributed to the possible embrittlement of grain boundaries, e.g. the presence of an embrittled layer due to a higher gas content (Table 5.6)[23]. The presence of some gross deformation at an improved impact toughness value of 42J is expected due to an improvement in overall ductility leading to some gross deformation (Fig. 5.18 B). Accordingly, the fracture appearence corresponding to the highest toughness value will reveal the existance of gross deformation in the form of shallow dimples, has been observed (Fig. 5.18C). The existance of secondary cracking in majority of fractographs is that an indication the material is not free of embrittlement.

The existance of gross deformation in the fractographs in fig. 5.18 D can be similarly explained.

Considering the fractographs of tensile tested speciment (Fig. 5.19 & 20; A to D), the existance of gross deformation in varying amount is due to reasonalbe level of %EL ( $\geq 12\%$ ) and also because tensile deformation mostly includes uniaxial loading, unlike the deformation under impact where biaxiality & triaxiality of the operating stress system is definitely involved. With an improvement is ductility/toughness the extent of gross deformation will increase as has been observed.

#### **CHAPTER-6**

# CONCLUSIONS AND SUGGESTIONS FOR FUTURE WORK

#### **6.1 CONCLUSIONS**

Under the existing experimental conditions, the following conclusions have been arrived at:

- i) Hardness in the as quenched conditions was a function of hardening temperature; the microstructure was accicular (low carbon martensite)
- ii) Hardness in the as tempered condition (tempering temp. 660°C & 685°C) was lower than the as quenched hardness and decreased marginally with tempering periods.
- iii)Microstructure in the as tempered condition retained the accicularity of the as quenched structure; a slight coarsening in it was observed at larger tempering periods.
- iv) The material behaved satisfactorily from the point of view of inclusion rating; however presence of chunky refractory/slag materials was an undesierable feature.
- v) Tensile properties is the as tempered condition were a function of quenching temperature as well as tempering temperature & time. In the 1010°C as quenched condition followed by tempering at 660°C & 685°C for periods ranging from 90 to 270 mins., PS ranged from 700 to 732 MPa, UTS from 814 to 875 MPa, % El from 14 to 22% & % RA from 60% 66% approximately. Under similar conditions, if a hardening temperature of 980°C was employed, the corresponding properties are 668 to 732 MPa, 776 to 862 MPa, 17 to 19.6% & 60 to 68% approximately. The inconsistancies in the data could be rationalised by

plotting the properties (as a function of dependent variables) using the best fit method. Taking an overall view, the tensile properties remained more or less unaltered on varying the tempering period.

vi) Impact toughnes improved overall with tempering temperature and less clearly with tempering periods. However, at the lower of the two hardening temperatures, the toughness in the as tempered condition showed a more distinctive tendency towards improvement both with tempering temperatures and periods. This has been attributed to favourable effect of a fine grain size.

vii) Fractographic appearance carried out on selected impact & tensile tested specimens was in general consistent with the overall state of the material.

#### **6.2 SUGGESTIONS FOR FUTURE WORK**

To make the appraisal more reliable and useful, the following testing regime should be followed.

a) From the point of view of mechanical properties, a detailed investigation relating to impact and tensile testing is required to be carried out. With the available and newly generated data, it would be possible to develop models for the structure-property relationships.

b) From the point of view of performance of the material for a particular application i.e. steam turbine blading, corrosion testing fatigue testing & wear testing is required to be carried out extensively.

c) Determination of the CCT diagram of the material.

d) Determination of the critical temperature.

## REFERENCES

- 1. Kodgire, V.D., "Material Science and Metallurgy for Engineers", EPH, 1994.
- 2. F.B. Pickenring, "The Metallurgical Evolution of Stainless Steels", ASM and The Metals Society (London), 1979.
- 3. Dr. B.R. Nijhawan and Mr. P.K. Gupta, "Symposium on Production Properties and Application of Alloy and Special Steels", NML, Jamshedpur, 1958.
- 4. Donald Peckner and I.M. Bernstein, "Handbook of Stainless Steels", Mc-Graw Hill Book Co., 1977.
- 5. Sedriks, J.C, "Corrosion of Stainless Steels", Corrosion Monograph Series, Wiley and Sons Inc., 1979.
- 6. Source Book on Stainless Steels, ASM, 1979.
- D.R. Holmes and R.B. Hill, "Corrosion of Steels in CO<sub>2</sub>", Proceedings of the British Nuclear Energy Society., 1974.
- 8. Metallurgical Developments in High Alloy Steels, Special Report of the Iron and Steel Institute, 1986.
- 9. Metals Handbook, Desk Edition, ASM, 1985.
- 10. N.R. Comins and J.B. Clark, "Specialty Steels and Hard Materials", Pergamon Press, 1982.
- 11. M. Columbier, "Stainless and Heat resistant Steels", Wiley & Sons Inc., 1978.
- 12. Metals Handbook, Vol. 1, 10th Edition, Properties and Selection: Iron, Steel and High Performance Alloys, ASM, 1990.
- 13. Prof. R.W.K. Honeycombe, "Steels, Microstructure and Properties", Metallurgy and Material Science Series, 1981.
- 14. L.L. Shreir, "Corrosion, Metals/Environment Reactions, Vol. 1, 1976.

- 15. DECHEMA, Corrosion Handbook, Vol. 1, Corrosive Agents and Their Interaction with Materials, 1987.
- 16. Juran's Quality Control Handbook, 4th ed., Mc-Graw Hill Book Co., 1988.
- 17. AISI 420, 1978.
- 18. AISI/AWS B70-77, 1978.
- 19. Metals Handbook, Ninth Edition, Vol. 9, Metallography and Microstructures, ASM, 1990.
- 20. ASTM E-23, 1978.
- 21. Roland Kiessling, "Non-metallic Inclusions in Steels", The Metal Society, London, 1978.
- 22. Metals Forum, Vol.2, No.2, 1979.
- 23. Personal communication with Dr. S.K. Goel.

# FIGURES & TABLES

Table2.1 : Compositions of the 300 and 200 series of Austenitic Stainless Steels.

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۸I! Grad	SI			(	Composit	ion <sup>b</sup> . 1	•			
Grad	de <u>Cr</u>	NI	C	Mn	51	P	S		Other	
301	16-11	96-8	0.15	2.0	1.0	0.045	5 0.03	n		
302	17-1		0.15	2.0	1.0	0.045				
3021			0.15	2.0	2-3	0.045				
303	17-19		0.15	2.0	1.0	0.20	0,15		Mo 0.60d	
3035			0.15	2.0	1.0	0.20	0.06		Se 0.15 <sup>C</sup>	
304 3041	18-20			2.0	1.0	0.045				
3041			0.03	2.0	1.0	0.045				
305	17-19		0.12	2.0 2.0	1.0,	0.045			N 0.10-0.1	5
308	19~2]		0.08	2.0	1.0 1.0	0.045				
309	22-24		0.20	-	1.0	- 0.045				
3095			0.08	2.0	1.0	0.045				
310	24-26		0.25	2.0	1.5	0.045				. •
3105	5 24-26		0.08	2.0	1.5	0.045				
314	23-26	5 19 -22	0.25	2.0	1.5-3.0					
316	16-18	10 -14	0.08	2.0	1.0	0.045			10 2.0 -3.0	
316F	16-18	3 10 -14	0.08	2.0	1.0	0.20	0.10	•	1.75-2.50	1
J161	, 16-18	8 10 -14	0.03	2.0	1.0	0.045			0 2.0 -1.0	
316N			0.08	2.0	1.0	0.045			2-3, N 0.1-0	).16
317	18-20		0.08	2.0	1.0	0.045			10 3.0 -4.0	
3171			0.03	2.0	1.0	0.045	0.030	) м	io 3.04.0	•
321	17-19		0.08	2.0	1.0	0.045	0.030	) т	L 5xC <sup>C</sup>	
330	17-20		0.08	2.0	0.75-1.			)		
347	17-19		0.08	2.0	1.0	0.045		) C	b+Ta l0xC	
348	17-19	9 -13	0.08	2.0	1.0	0.045	0.030	) C	D+Ta 10xC <sup>c</sup> 0.1; Co 0.	
384	15-17	17 -19	0.08	2.0	1.0	0.045	0.030	Та	0.1 <sup>-</sup> ; Co 0	. 20°
AISI			c	ompos	Ition <sup>a</sup> .	1				
Grade	Cr	NI	C	Mn	51	<del>.</del>	Ś	11	Other	
201	16 -18	3.5-5.5	0.15 5	.5- 7,	5 1.0	0.060	0.030	0.25	-	
202	17 -19	4-6	0.15 7	.5-10	1.0	0.060	9.030	0.25	-	
205	16.5-18	1-1.75	0.25	14-15	.5 1.0	0.060	0.030	0.40	1-1.75Mo	
16 <sup>b</sup>	17.5-22	5-7	0.08 7	.5-9	. 1.0	0.045	0.030	0.50	3Mo	
		5.				0.015	0.050		5110	
16L <sup>b</sup>	17.5-22	5-7	0.03 7	.5-9	. 1.0	0.045	0.030	0.50	ЗМо	
•	•									
Balan	ce iron.	Single va	luos are	mayin	nim valu	os unlas	e other	معات	noted.	
Not s	tandard A	Single va ISI grades					o ounea			
ource	s: Sourc	e Book on	Stainles	s Stee	els, Ame	rican So	cietv :	or Me	tals,	
-	1976						, .			
		heny Ludlu	m Alloy	Data.		. •				
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# Table2.2 : Mechanical properties of the 300 and 200 series of Austenitic Stainless Steels.

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AISI	Tensile Strength	Yield Strengt (0.21 Offset)		n, Hardness,
Grade	MPa	MPa	1	Rockwell B
	365			
301	758	276	60	85
302	620		50	85
302B	655	276 241	55	85.
303 <sup>5</sup> 303Se <sup>b</sup>	620 620	241	50 50	76
30350	586	- 241	55	80
304 304L	517	193	55	79 -
304L	620	331	50 .	85
3041	586	262	50 .	80
308	586	241	50	80
309	620	310	45	85
3095	620	310	45	85
310	655	310	45	85
310S	655	310	45	85
3103	690	345	40	85
316	620	276	50	79
316F	586	241	60	85
316L	517	220	50	79
316N	620	331	48	85
317	620	276	45	85
317L	586	241	55	85
, 11/5	620	241	45	80
321 330b	586	310	40	. 80
347	655	276	45	85
348	655	276	45	85
384	517	241	55	70
504	517			• •
	- ···	Yield		
	Tensile	Strength		
AISI	Strength	(0.23 Offset)	Elongation	Hardness,
Grade	<u>MPa</u>	MPa		Rockwell B
201	758	379	55	90
202	689	379	55	90
205	862	482	58	9 B
216 <sup>C</sup>	689	379	45 ·	9 Ż
216L <sup>C</sup>	689	379	45	92
				1

a Annealed sheet and strip. bl MPa = 145.03 psi. CNot standard AISI grades. Sources: Source Book on Stainless Steels, American Society for Metals, 1976. Allegheny Ludium Alloy Data.

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Table2.3 : Compositions of some Austenitic Ni-Cr-Fe Alloys.

			тур_	ical Compos	ition <sup>a</sup> , (1)	
Alloy	<u>N1</u>	Cr	Fe	C	Co	TI
AISI Type 304 <sup>b</sup>	10	19	69	0.06	0.15	0.008
Alloy 800 <sup>C</sup>	33	21	38	0.03 max.	0.10 max.	0.60 max. <sup>f</sup>
INCONEL Alloy 600 <sup>d</sup>	76	15	8.	0.04	0.03	0.25
INCONEL Alloy 690 <sup>e</sup>	60	30	9.5	0.03	0.03	0.25

Table 2.4 : Compositions of some Austenitic Ni-Cr-Fe-(Mo, Cu, Cb) Alloys.

		т	ypical	Compo	sition	(Nt.	13	
Alloys	NI	Cr	<u>Fe</u>	<u>No</u>	C	<u>Cb</u>	<u>ču</u>	Other
AISI Type 317	13.0	19.0	64.0	3.5	0.05	'		<b></b> `
SANDVIK 2RN65	24.0	17.5	46.0	4.7	0.02			
SANDVIK 2RK65	25.0	19.5	50.0	4.5	0.02		1.5	
AL-6X	25.0	20.0	48.5	6.5	0.01			
JESSOP 700	25.0	20.0	49.0	4.4	0.02	~~		
JESSOP 117	25.0	21.0	45.0	4.6	0.02	0.3	2.5	
UDDEHOLM 904L	25.0	20.0	49.0	4.5	0.92		1.5	
HAYNES No. 20 (Mod)	26.0	22.0	45.0	5.0	0.23	-,-	~ ~	Ti 4×C
CARPENTER 20Cb-3	33.0	20.0	41.0	2.0	0.03	0.8	1.0	,
INCOLOY Alloy 825	42.0	20.0	30.0	3.0	0.03		2.0	<b>~~</b> .
HASTELLOY Alloy G	45.5	22.0	19.5	6.5	0.03	2.0	2.0	W 0.5
HASTELLOY Alloy C-276	59.0	16.0	5.0	16.0	0.02			W 4.0
INCONEL Alloy 625	62.0	22.0	3.0	9.0	0.05	3.5		
	•							

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Table 2.5 : Compositions of the 400 series of Ferritic Stainless Steels.

ade		Cr	C	Mn	SI	P	<u>tlon<sup>a</sup>, (</u>	Other
05 <sup>.</sup>	11.9	5 -14,5	0.08	1.0	1.0	0.040	0.030	A1 0.10-0.30
09	10.9	5 -11.7	0.08	1.0	1.0	0.045	0.045	ті 6×С <sup>d</sup> ,0.75 <sup>C</sup>
9.	14	-16	0.12	1.0	1.0	0.040	0.030	
0	16	-18	0.12	1.0	1.0	0.040	0.030	
30F	16	-18	0.12	1.25	1.0	0.060	0.15 <sup>d</sup>	Mo 0.6 <sup>e</sup>
FSe	16	-18	0.12	1.25	1.0	0.060	0.060	. Se 0.15 <sup>d</sup>
4.	16	-18	0.12	1.0	1.0	0.040	0.030	Mo 0.75-1.25
6	16	-18	0.12	1.0	1.0	J.04D	0.030	Cb+Ta 5xC <sup>d</sup> , 0.7 <sup>c</sup> , No 0.75-1.25
9 <sup>b</sup>	17.7	15-18.75	0.07	0.60	0.60	0.040	0.030	Ti 12xC <sup>d</sup> ,1.0 <sup>C</sup> ,Ni 0.5
2	18	- 2 3	0.20	1.0	1.0	0.040	0.030	
6	23	- 27	0.20	1.5	1.0.	9.040	0.030	N 0.25

datance from. Single values are a c 439 is not a standard AISI grade. d Maximum. C Optional.

Source: Source Book on Stainless Steels, American Society for Metals, 1976.

Table 2.6 : Mechanical properties of the 400 series of Ferritic Stainless Steels.

AISI Grade	Tensile Strength 	Yield Strength MPa	Elongation, 1	llardness, Rockwell B
405	482	276	30	<b>8</b> 0 ·
409	469	276	2 5	75
429	486	293	30	76
430	517	310	30	82
430F	551	379 ,	25	86
430FSe	551	379	25	86
434	531	365	23	83
436	531	J65	23	83
4 ] 9 <sup>C</sup>	482	280	30	80
442	551	345	20	90
446	551	345	25	86

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aAnnealed material. bl MPa = 145.03 psi. C439 is not a standard AISI grade. Source: Source Book on Stainless Steels, American Society for Metals, 1976.

	ion <sup>a</sup> , (%) Other	ł	1	NÍ 1.25-2.50	Mo 0.60 <sup>d</sup>	se o.15 <sup>b</sup>	1	ма 0.60 <sup>d</sup>	Ni 0.50-1.0,45 0.75-1.25,8 9.75-1.25,V 0.15-0.30	Ni 1.25-2.50	MO 0.75	Mo 0.75	110 0.75	S 0.08 or Sc 0.18	iron. Single values are maximum values unless otherwise noted. dard grade, typical composition. Source Book on Stainless Steels, American Society for Metals, 1976.
	Composition <sup>a</sup> , S	0.030	0.030	0.030	0.15 <sup>b</sup>	0.060	0.030	0.15 <sup>d</sup>	0.025	0.030	0.030	0.030	0:030	ł	s unless can Soci
		0.040	0.040	0.040	0.060	0.060	0.040	0.060	0.025	0.040	0,040	0.040	0.040	0.04	m value: , Amcri
	SL	0.5	1.0	1.0	1.0	1.0	1.0	1.0	0.75	1.0	1.0	1.0	1.0	0.4	maximu sition Steels
1	Чи	1.0	1.0	1.0	1.25	1.25	1.0	1.25	1.0	1.0	1.0	1.0	1.0	0.4	s are compo nless
	U	0.15	0.15	0.15	0.15	0.15	0.15 <sup>b</sup>	0.38	0.20-0.25	0.20	0.60-0.75	0.75-0.95	0.95-1.20	1.0	Single values are maximur grade, typical composition. e Book on Stainless Steels,
	Cr	. 5-13	11.5-13.5	11.5-13.5	-14	-14	-14	-14	-13	-17	-13	-18	-18		<sup>a</sup> Balance iron. <sup>9</sup> bhinimum. c <sup>N</sup> on-standard gra Optional. Source: Source 1
		11	11	11	12	12	12	12	11	15	16	16	16	17	nce mum. star onal
	AISI Grade	403	410	414	416	416Se	420	420F	422	43I	440A	4408	440C	440F <sup>C</sup>	a Balance Mainimum CNon-sta doptiona Source:

49

÷.

Table 2.7 : Compositions of the 400 series of Martensitic Stainless Steels.

Table 2.8 : Machanical	properties	of the 400	series of M	Aartensitic	Stainless Steels.
------------------------	------------	------------	-------------	-------------	-------------------

AISI Grade		Tensile Strength <u>MPa</u>	Yield Strength (0.2% Offset) NPa	Elongation, 1	Hardmess, Rockwell D or C
403,410,	۸ <sup>b</sup>	517	276	30	E 32
416,4165e	H <sup>C</sup> +T at 205°C	1310	1000	15	C 41
410, 12000	316°C	1241	965	15	C 39
	538°C	1000	793	20	C 31
-414	A	827			
• • •	IIC+T at 205*C	1379	1034	15	C 43
	316°C	1310	1000	15	C 41
	538°C	1000	827	20	C 34
420,4205		655	345	25	3 92
	Hd +T at 316 °C	1586	1345	8	C 50
422	· · · · · · · · · · · · · · · · · · ·	793	586	22	3 98
	11 <sup>C</sup> +T at 427*C	1627	1282	10	
	5 J 8 ° C	1476	1145	· 13	· C 42
	649°C	1000	862	14	C 32
431		862	655	20	<u> </u>
	и <sup>d</sup> +таt 205°C	1413	. 1069	15	C 43
	316°C	1345	1034	15	J 41
	538°C	1034	896	18	- C 34
4407		724	414	20	2 95
	н <sup>d</sup> +т аt 316•с	1793	1655	5	C 51
4408	, <u> </u>	738	429	18	2 96
	и <sup>d</sup> +т ав 316°C	1930	1862	3	C 55 -
440C, 440F	Λ.	758	448		<u> </u>
• • •	Hder at 316"C	1965	1895	2	2 57

a Non-standard grade. bA = annealed. cII = hardened by heating to 982°C and cooling. dII = hardened by heating to 1038°C and cooling. T = tempered at indicated temperature. Source: Metals Handbook, American Society for Metals, Vol. 1, 1969.

Table 2.9 : Compositions of some Duplex Stainless Steels.

		_	T	ypical	Comp	ositio	n <sup>a</sup> , (1	.)	
Designation	Cr	NI	<u>_</u> C	Nn	<u>si</u>	<u>P</u>	S		Other
AISI Type 329	28.0	6.0	0.10	2.00	1.0	0.04	0.03	Мо	1.5
326 <sup>b</sup>	26.0	6.5	0.05	1.00	0.6	0.01	0.01	Тİ	0.25
SANDVIK JRE60	18.5	4.5	0.02	1.50	1.6	0.01	0.01	Мо	2.5
a Balance iron. Earlier versic available sinc AL 326 and H-3 Sources: Varic	e 1970 26.								

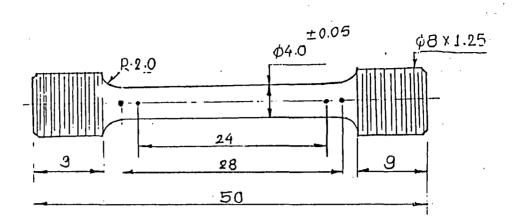
Table 2.10 : Mechanical properties of some Duplex Stainless Steels.

_Designation	Tensile Strength MPa	Yicld Strength (0.2% Offset) MPa	Elongation,	Hardness, Rockwell B
AISI Type 329	724	551	25	. 98
326	689	517	35	95
SANDVIK 3RE60	717	482	48	9 2

a<sub>M</sub>ill annealed condition. b MPa = 145.03 ksi. Sources: Various.



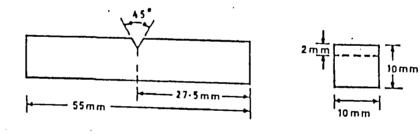
۶n

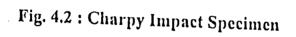


# Fig. 4.1 : Tensile Specimen

(All dimesions are in mm)

Scale 1:2





### TABLE 5.1

# INSTRUMENTAL ANALYSIS FOR X20Cr13 GRADE STEEL

Alloying	Cr	Ni	C	Mn	Si	S	Р
Elements							
Allowable	12.5-14	0.3-0.8	0.17-0.22	0.3-0.8	0.1-0.5	0.02	0.03
%	:					max	max
Actual	13	0.40	0.20	0.50	0.30	0.017	0.027
%							

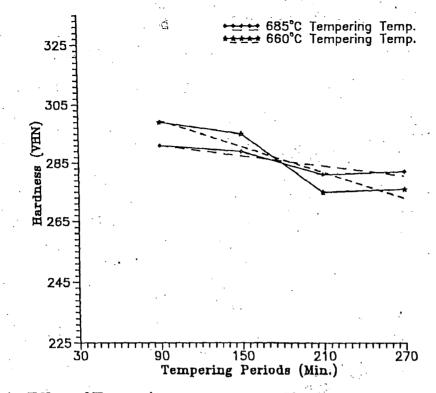
#### **TABLE 5.2**

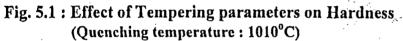
S. No.		Heat Treatmer	Hardness HV <sub>30</sub>	Standard Deviation σ	
A		$C + 30 \min(\text{soak})$		512 460	11
В	Hardening	C + 30 min (soaki Tempering	Tempering	400	9
	Temp. (°C)	Temp. (°C)	Periods(Min.)		
1	1010	685	90	291	12
2		-	150	289	14
3		-	210	281	10
4			270	282	15
5		660	90	299	9
6			150	295	12
7			210	275	16
8			270	276	14
9	980	685	90	296	8
10			150	286	16
11			210	265	12
12	,		270	265	10
13		660	90	287	15
14			150	292	9
15	· ·		210	269	11
16			270	267	16

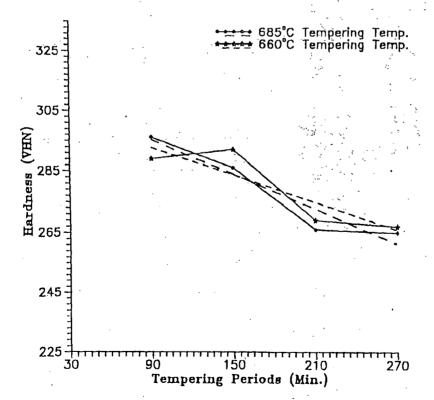
# EFFECT OF HEAT TREATMENT ON HARDNESS (Hardness of as Rolled Experimental Slap: $282 \text{ HV}_{30}$ )

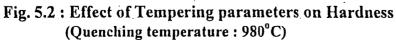
\* Soaking period for hardening prior to tempering is 90 minutes, followed by oil quenching.

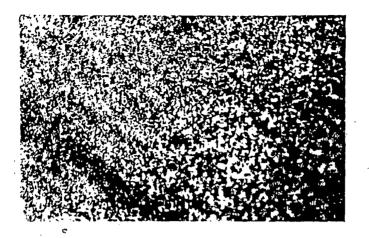
\* Tempering treatment is followed by Air Cooling.











(A)



(B)

Fig. 5.3 : Effect of Heat Treatment on Microstructure. (A, B represent microphotographs taken at 100X and 400X magnification respectively, in as quenched condition. Quenching temperature : 980°C.)

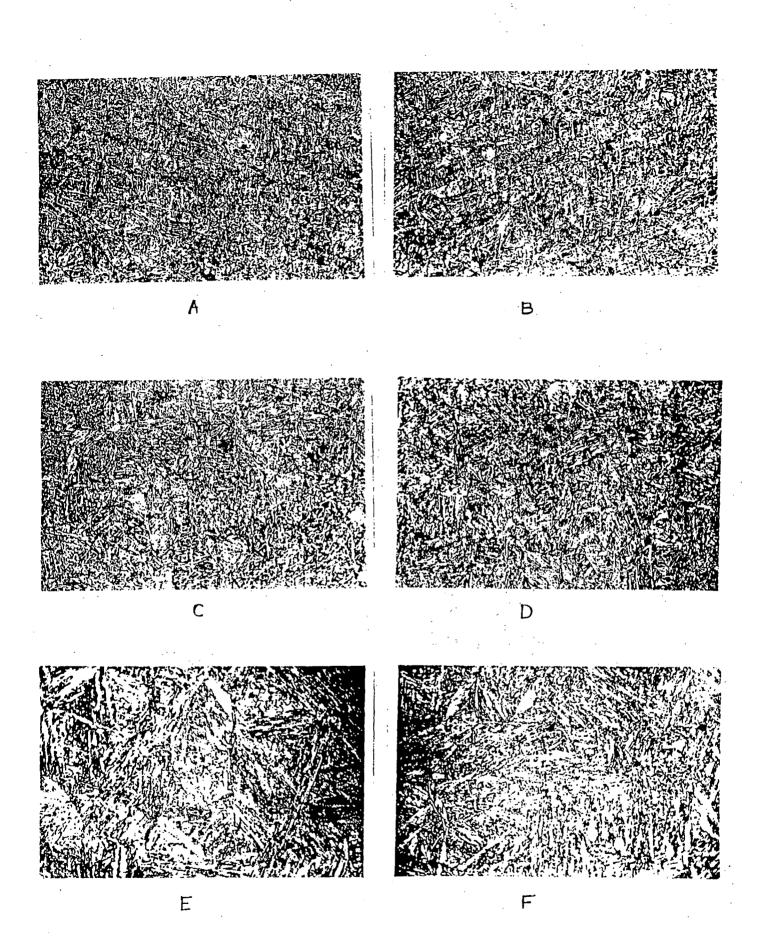


Fig. 5.4 : Effect of Heat Treatment on Microstructure.
(A, B, C, D) represent microphotographs taken at 100X magnification of 1,4,5,8th specimens respectively. E,F represent microphotographs taken at 400X magnification of 4,8 Nos. specimens respectively)

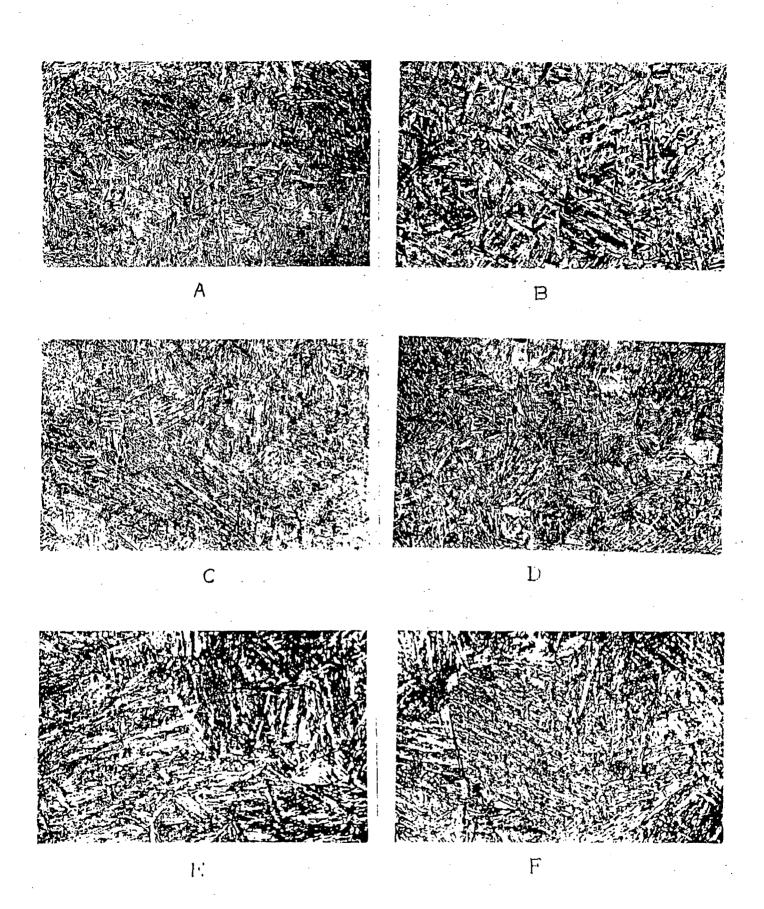
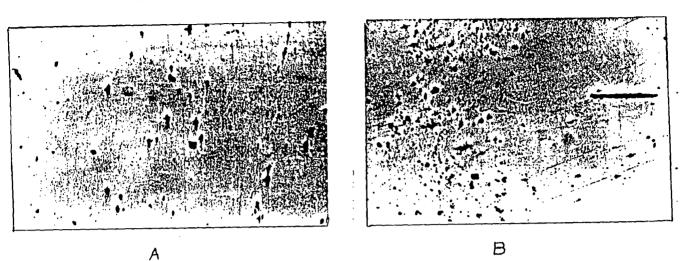
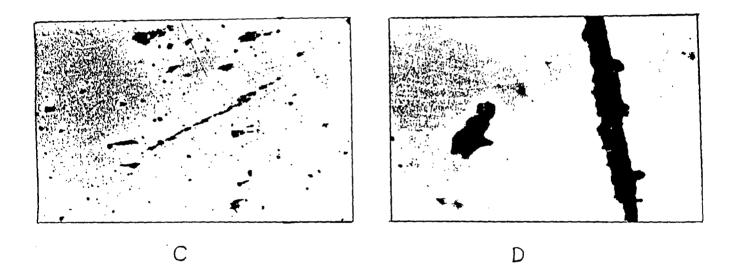


Fig. 5.5 : Effect of Heat Treatment on Microstructure. (A, B, C, D represent microphotographs taken at 100X magnification of 9,12,13,16 Nos. specimens respectively. E, F represent microphotographs taken at 400X magnification of 9, 13 Nos. specimens respectively)





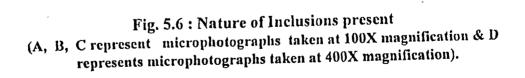


Table 5.3 A : Micro Probe	Analysis of Non Metallic Inclusions present in X20Cr13
Grade Steel.	

				· .		
No.	3 Name	: 5-P1				
Element SiO2 Al2O3	Wt. (%) 0.680 0.011	to 100% 0.640 0.010	+/- 0.073 0.063	Cation 0.6597 0.0121	K-ratio 0.598 0.008	
. FeO	59.357	55.837	0,407	48.1270	62.471	
MgÜ	0.132	0.124	0.096	0.1902	0.083	
MnO	0.592	0.557	0.073	0.4860	0.633	
CaO	0.356	0.335	0.024	0,3698	0.380	
K20	2.251	2.117	0.042	2.7841	2.487	
Na20	6,065	5.706	0.350	11.4020	3.069	
Ti02	0.046	0.044	0.033	0.0338	0.055	
Cr203	36.814	34.631	0.793	28.2191	44.291	
Total	106.304	100.000		92.2839	114.073	

Table 5.3 B : Micro Probe Analysis of Non Metallic Inclusions present in X20Cr13Grade Steel.

Ño.	4 Name	: ,,-P2			
Element SiO2 Al2O3 FeO MgO MnO CaO K2O Na2O TiO2 Cr2O3	Wt. (%) 2.212 0.009 98.631 0.153 1.090 0.006 0.000 0.000 0.000 60.902	to 100% 1.357 0.005 60.509 0.094 0.669 0.003 0.000 0.000 0.000 0.000 37.363	+/- 0.156 0.126 0.524 0.182 0.093 0.021 0.000 0.000 0.000 1.012	Cation 1.3799 0.0065 51.4539 0.1422 0.5760 0.0037 0.0000 0.0000 0.0000 30.0365	K-ratio 1.946 0.007 104.287 0.096 1.179 0.006 0.000 0.000 0.000 74.229
Total	163.003	100.000		83.5986	181.743

#### TABLE 5.4

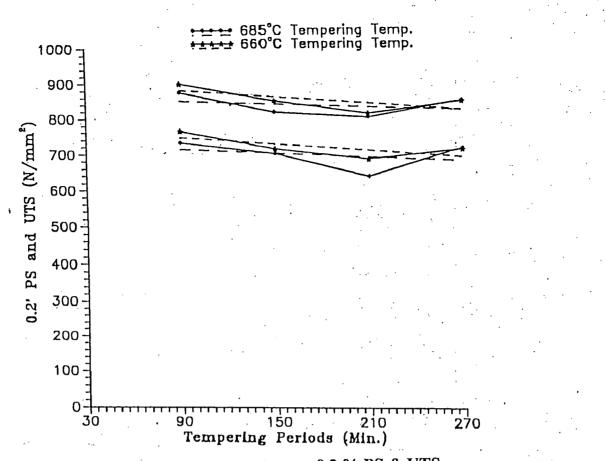
			· · · ·						· ·	·	
					6 P.S.	UT		EL %		R.	
S. No.	Heat Treatment		at .	(Mpa)		Мра		%		76	
5.110.	Hardening Temp. (°C)	Tempering Temp. (°C)	Tempering Periods(Min.)	1*	2**	1*	2**	1*	2**	1*	2**
1	1010	685	90	732	710	875	873	17.15	17.68	65.00	66.60
2			150	701	676.4	818	820	20.83	19.80	66.97	68.75
3			210	637	683	803	854	14.20	17.70	60.00	66.60
4			270	716	666	851	852	16.67	17.92	66.25	64.63
5		660	90	764	741	899	904	15.83	17.08	67.50	70.37
6			150	714	643	850	802	17.50	20.42	64.19	65.85
7		-	210	685	669	814 -	796	19.60	17.50	61.25	68.35
8			270	712	730	848	832	22.10	18.59	64.63	68.67
9	980	685	90	732	714	851	883	17.50	18.75	57.50	67.90
10			150	667	676	811	859	. 19.20	19.60	60.49	68.75
11	i .		210	637	620	780	780	19.60	17.92	59.25	60.00
12			270	637	636	776	784	19.60	18.75	66.60	59.75
13	-	660	90	730	721	862	842	19.20	17.50	66.60	59.25
•14			150	699	713	838	860	17.92	20.00	66.60	59.25
15			210	683	644	818	893	17.10	17.30	64.19	66.66
16			270	668	692	822	831	18.75	16.67	60.49	60.00

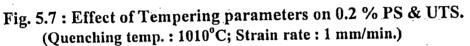
# EFFECT OF HEAT TREATMENT ON TENSILE BEHAVIOUR

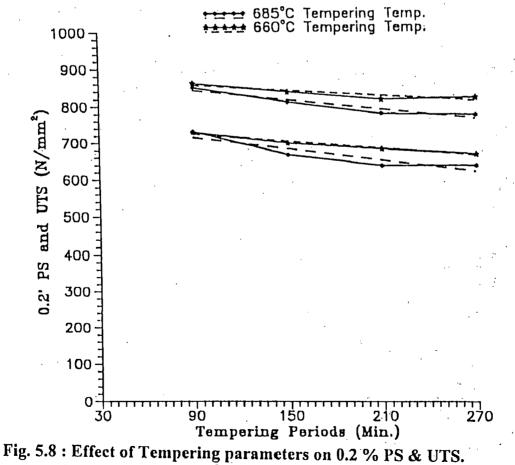
- Soaking period for hardening, prior to tempering is 90 min., followed by oil quenching.

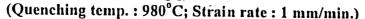
- Tempering treatment is followed by Air Cooling.

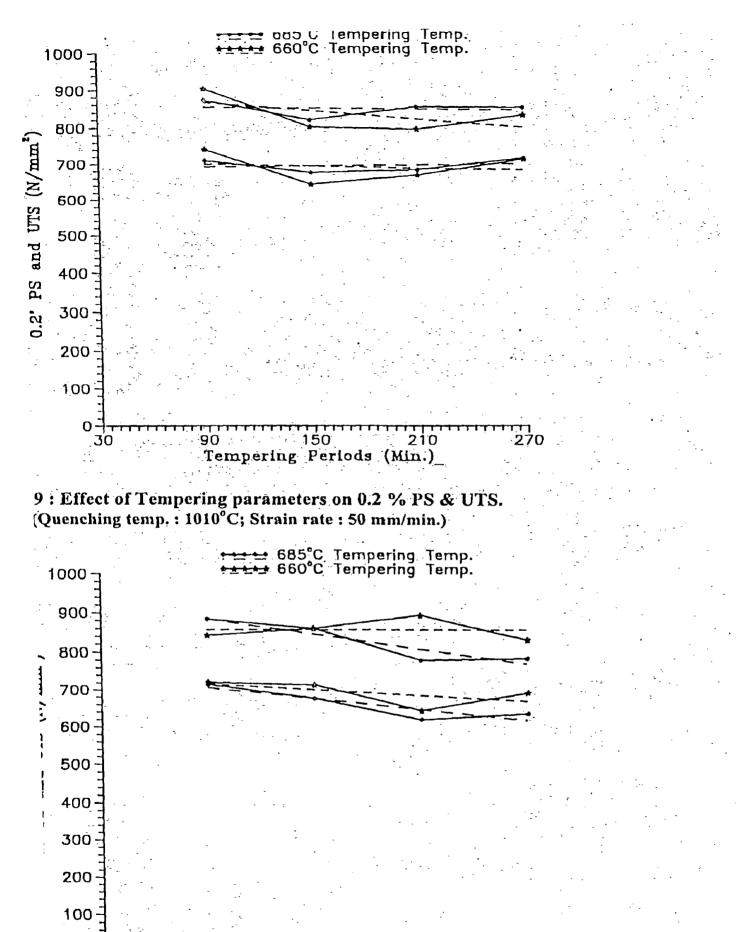
- \* 1 =Strain rate 1 mm/min
- \*\* 2 = Strain rate 50 mm/min

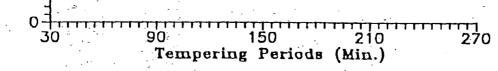




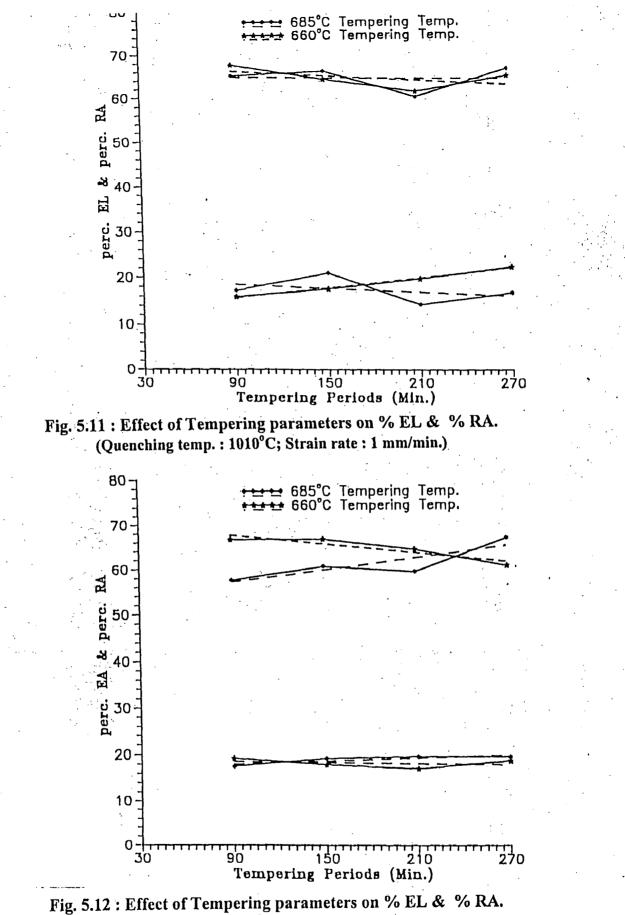


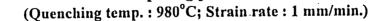


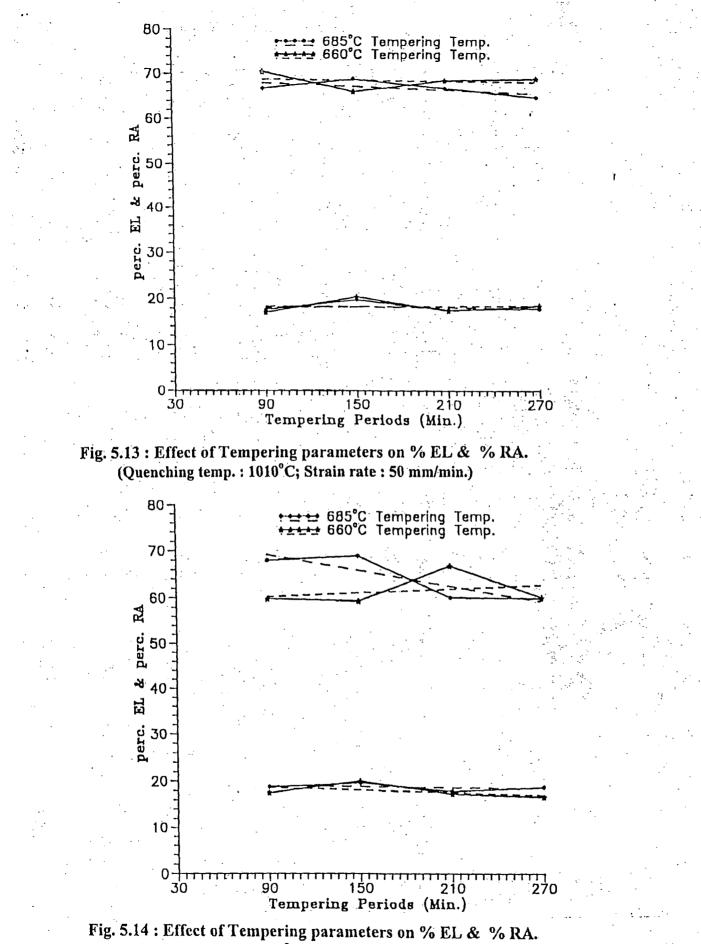


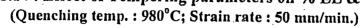


0 : Effect of Tempering parameters on 0.2 % PS & UTS. (Quenching temp. : 980°C; Strain rate : 50 mm/min.)







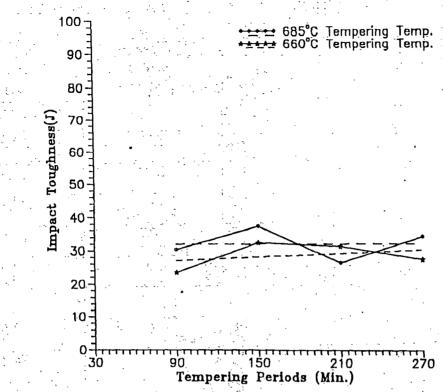


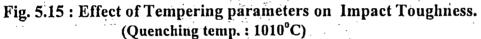
**TABLE 5.5** EFFECT OF HEAT TREATMENT ON IMPACT TOUGHNESS

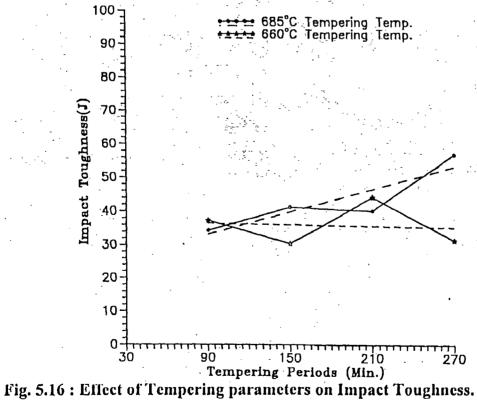
Specimen	Hardening	Tempering	Tempering	Impact
(Block no.)	Temperature	Temperature	Periods	Toughness
-	(°C)	(°C)	(Min.)	(J)
1	1010	685	90	30.4
2			150	37.3
3			210	26.5
4			270	34.3
5		660	90	23.5
6		· ·	150	32.4
7			210	31.4
8		· · · · · · · · · · · · · · · · · · ·	270	27.5
9	980	685	90	34.3
10	:		150	41.2
11			210	40.2
12			270	56.9
13		660	90	37.3
14	· .		150	30.4
15		,	210	44.2
16			270	31.4

\* Soaking period for hardening, prior to tempering is 90 min., followed by oil quenching.
\* Tempering treatment is followed by Air Cooling.

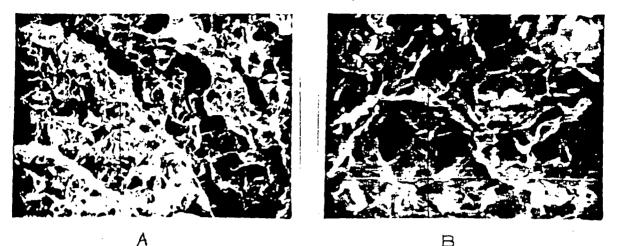
\* Remark - Samples broken in all the cases.







(Quenching temp. : 980°C)



в

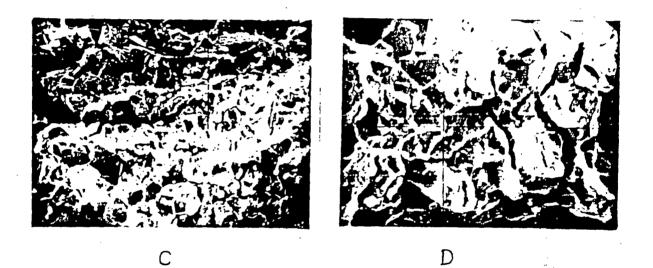
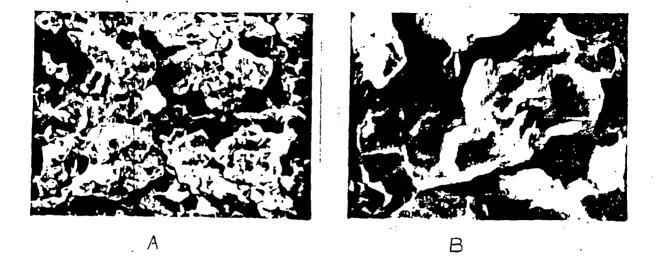
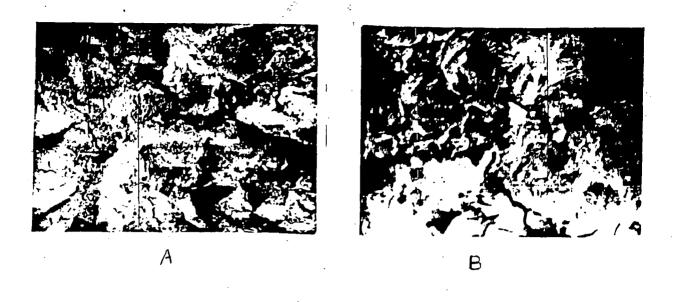


Fig. 5.17 : Fractographic behaviour of Impact Specimens. (A,B,C,D represents SEM photographs of specimens no. 1, 3, 5 and 6 at magnifications 76X, 152X, 76X and 152X respectively).



C D

Fig. 5.18 : Fractographic behaviour of Impact Specimens. (A,B,C,D represents SEM photographs of specimens No. 9, 10, 12 and 15 at magnifications 72X, 200X, 144X and 216X respectively).



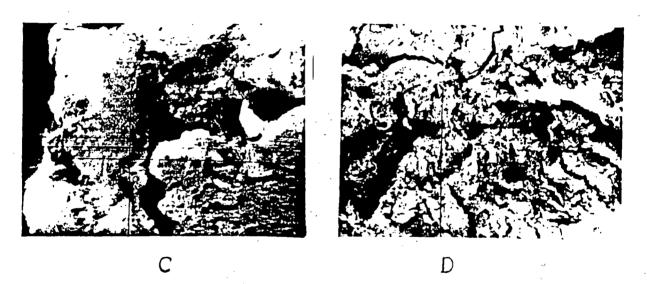
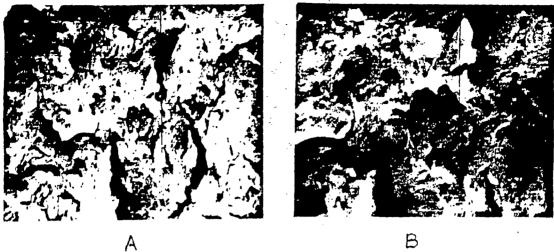


Fig. 5.19 : Fractographic behaviour of Tensile Specimens. (A,B,C,D represents SEM photographs of specimens No. 1,4,5 and 8 at magnifications 56X, 112X, 104X and 60X respectively).



А

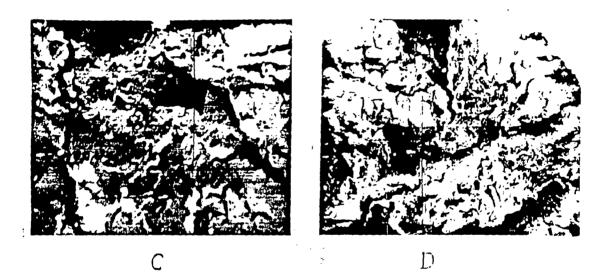


Fig. 5.20 : Fractographic behaviour of Tensile Specimens. (A,B,C,D represents SEM photographs of specimens No. 9,12,13 and 16 at magnifications 120X, 240X, 104X and 56X respectively).

# GAS ANALYSIS FOR $\mathrm{O}_2$ AND $\mathrm{N}_2$ IN X20Cr13 GRADE STEEL

SPECIMEN NO.	O <sub>2</sub> (parts per million)	N <sub>2</sub> (parts per million)
1	47.2	317
2	51.9	314