APPRAISAL OF AN AUSTENITIC STEEL IN THE AS-ROLLED/SEMI-ROLLED CONDITION

A DISSERTATION

submitted in partial fulfilment of the
requirements for the award of the degree
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MASTER OF ENGINEERING
in
METALLURGICAL AND MATERIALS ENGINEERING
(With Specialization in Industrial Metallurgy)

By

NEERAJ NAUTIYAL

DEPARTMENT OF METALLURGICAL AND MATERIALS ENGINEERING
UNIVERSITY OF ROORKEE
ROORKEE-247 667 (INDIA)

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CANDIDATE'S DECLARATION

I hereby declare that the work presented in this dissertation entitled **"APPRAISAL OF AN AUSTENITIC STEEL IN THE AS ROLLED/SEMI-ROLLED CONDITION"** in partial fulfillment of the requirements for the award of the degree of **Master of Engineering** in Metallurgical and Materials 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 1998 to March 1999 under the guidance of **Dr. A.K. Patwardhan**, Professor, 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.

Dated : March 26, 1999
Place : Roorkee

(Neeraj Nautiyal)

CERTIFICATE

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

A. K. Patwardhan
(A.K. Patwardhan)
Professor
Deptt. of Metallurgical & Materials Engg.
University of Roorkee,
Roorkee - 247 667 (U.P.)

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MY THIS PIECE OF WORK IS DEDICATED TO MY FATHER.

Nautiyal
(Neeraj Nautiyal)

ABSTRACT

An experimental study has been made of the effect of heat treatment on the mechanical properties of austenitic grade of stainless steel used in automobile application.

The present investigation was carried out in the form of material/product appraisal. It was observed that hardness was a function of heat treating temperature, time and cooling rates. The desired level of hardness ~ 30 - 32 Rc can be arrived at through a number of heat treating cycles.

The microstructure of the material consists of austenitic matrix and dispersed second phase namely carbides and nitrides. The nature of carbide to form was a function of heat treating temperature and time.

The material behaviour, from the point of view of inclusion rating and elemental segregation was acceptable.

The tensile properties stipulated in the standards namely hardness ~32 HRc, 0.2% P.S. ~ 580 MPa, UTS ~950-1200 MPa, % El ~8, % RA ~ 10 could be obtained through various combinations of solutionzing and ageing treatments, the optimum heat treatment being solutionizing from 1150^{0}C followed by ageing at 780^{0}C for 10 hrs.

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

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Chapter – 1

INTRODUCTION

Steels are used for a wide variety of conditions entailing heat & corrosion under both static & dynamic stresses. The behavior of metal changes significantly as their temperature is raised. At high temperatures under mechanical stress, metal undergoes plastic deformation, which continues as a function of time, this phenomenon is known as creep. At the same time, metal exposed to high temperature are much more liable to corrosion attack by any media with which they may be brought into contact & this phenomenon is known as high temperature corrosion or scaling.

Stainless steels are alloys containing a minimum of 11% chromium. Corrosion resistance is the most important of their properties. 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 ≥ 12% also provides useful oxidation resistance. Hence, their versatility lies in their being used as materials to resist corrosion and high temperatures.

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. Some of the grades contain chromium approaching 30%. 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 and applications. The grade 19 Cr-Mn-Ni, which is the experimental material in the present study is particularly useful for exhaust valve particularly in automobiles. This type of Stainless steel should have a high temperature resistance against creep and oxidation.

The main objective of the present study is (i) to assess whether the experimental material meets with the requirements as required by international standards (ii) to suggest any changes/modification that may be required to be carried out in the designing, manufacturing and processing practices to improve upon the expected level of performance. The various tests performed for the appraisal are tensile and standard optical metallography, macro etching , EPMA, SEM, X-RD 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. 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 chapter4. Experimental results are summarized and discussed in chapter 5. Conclusions and suggestions for future work are given in chapter- 6.

CHAPTER-2

LITERATURE REVIEW

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 steel are iron alloys containing a minimum of approximately 11% Cr. This amount of Cr, prevent 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 the minimum chromium content 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,

Cr in solid solution form = Total Cr - (17 x % 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 compositions 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], and 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. Type 305 and 384, which have higher nickel contents, exhibit low work hardening rates and are used in application 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. Type 316 and 317 contain molybdenum and have a greater resistance to pitting in marine and chemical industries. Type 321, 347 and 348 are grades that are used for applications requiring welded structures for elevated temperature service. [5].

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 up to 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 application [6].

Table 2.4 gives the typical compositions of Ni-Cr-Fe-(Mo, Cu, Cb) alloys. Out of these, Inconel alloy 625 and Hastelloy 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 ferritic 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 430 F and 430Fse, have also been popular to some extent [5].

In type 405, the addition of approximately 0.2% Al, 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 of 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 ^{0}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 mechanical properties (e.g., high strenght with adequate toughness, corrosion plus abrasion resistance, good resistance after heat treatments fatigue resistance after heat treatments, Hardenability throught 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.

1. Martensitic type precipitation hardening is achieved by simple ageing treatment of fabricated part. The precipatition hardening process is thought to involve the formation of very fine intermetallics. Prolonged aging causes these intermetallics, to coarsen, enabling the dislocations, to by pass 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 slight reduction of corrosion resistance and an increase in susceptibility to hydrogen embrittlement [9].

2. The semi-austenitc 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 ^{0}C to reduce the carbon and chromium contents of the austenite by the precipitation of carbides. [9].

3. 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 austenitc and ferritic 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 SUB CLASSIFICATION OF AUSTENITIC STAINLESS STEELS

The austenitic steels used for their scaling resistance fall into three categories : 20/10, 25/12, 25/20. Steels

2.3.1 20/10 Chromium – Nickel Steel (11)

The 20/10 Steels are quite similar to the 18/10, grades have carbon content from 0.03%/ 0.06% /0.20% for low, medium & high carbon content respectively & Ni up to 10 to 12%. Their resistance to scaling is satisfactory upto 900 ^{0}C in oxidizing conditions. The austenitic steels offer certain advantage over ferrite steels at same Cr content, and are easy to fabricate (by welding in particular) & their higher mechanical strength at elevated temperature.

The 20/10 steels are very extensively used for annealing boxes, recuperators, furnace components, tuyeres, exhaust ducts etc.

2.3.2 25/12 Chromium – Nickel Steel (11)

The 25/12 steels will withstand oxidizing conditions at temperature up to 1150 ^{0}C; if sulphur is present and the conditions are reducing, the ceiling temperature is down to 900 ^{0}C. They are very similar in properties to the 20/10 grades.

They are used for carbonizing and annealing boxes, a variety of furnace components & salt bath crucibles. The main advantage over the 20/10 grades is that the ceiling temperature in oxidizing atmosphere is about 200 ^{0}C higher.

2.3.3 25/20 Chromium – Nickel Steel (11)

The 25/20 grades are among the most used. They often contain 1-2% Si and resist scaling up to 1150 ^{0}C in ordinary oxidizing atmosphere, 1150 ^{0}C in the presence of small amounts of sulphur (up to 3g/m3) but only 900 ^{0}C if the sulphur concentration is high under these circumstances, the ferritic 28% Cr grades are to be preferred.

Their resistance to thermal fatigue & temperature cycling is good, they are similar in this respect to the 20/10 steels, better than 25/12 grades & in particular, greatly superior to the ferritic steels.

The following value applies to all three categories :-

Temperature, ^0C	700	800	900	1000	1100
1000 hr. creep – rupture stress, tsi.	5	3	2	1.1	0.4
1 % Permanent strain in 1000 hr. tsi.	4	2.2	13	0.8	0.25

Their mechanical strength & creep properties are much the same as for the other austenitic steels.

The 25/20 steels have very numerous uses, including super heater supports, heat exchangers & recuperatures components for boiler hearths & furnaces, carburizing & annealing boxes, salt bath crucibles & combustion chamber for gas turbines. They are used as forging, plate fabrication & castings.

The Figure 2.1 shows the creep rupture stresses for a 30-minute life at different temperature, for different austenitic steels, clearly revealing the superiority of 25/20 grades on others.

2.3.4 Miscellaneous Austenitic Steels

Some austenitic alloy with less than 30% Ni, & still many specific grades of steel has been developed for high scaling resistance. A particularly note worthy example is the group of steels developed for the manufacture of exhaust values for I. C. engines.

The basic advantage of austenitic valve steels is their high strength at elevated temperature & their consequent freedom from distortion troubles. A typical steel contains 0.4-0.5% C, 13-15%Cr, 13-15%Ni & 2-3% W. After treatment at 1050-1150 °C this steel given the following properties:-

Yield point	29-35 tsi
Tensile Strength	51-57 tsi
Elongation	30-40%

Key hole Charpy Impact test 10 kg/cm2.

Its tensile strength at high temperature is well above those of the martensitic Cr- Si valve steels . The data on short term tensile test at elevated temperature are given , in table 2.12

The curves in fig. 2.2 relates to 25 minutes creep rupture tests and again demonstrates the superiority of the austenitic steel.

Valves can also be manufactured from steels with higher chromium contents, to improve their scaling resistance, the austenitic structure is retained by balancing additions of nickel, Mn & nitrogen .

Table 2.13 lists a number of these steels . Type B in particular (21/4/N) has been adopted by the automobile industry . The table 2.14 lists the average mechanical properties of these steels , at R.T. & 750 °C , after quenching in water from 1175 °C & tempering for 12 hours at 750 °C .

This group of steels offers a useful range of combination between scaling resistance & creep strength . They all have much the same resistance to oxidation.

2.4 EFFECT OF ALLOYING ELMENTS ON THE PROPERTIES OF STAINLESS STEELS

Presence of alloying elements helps 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, while % elongation decreases, with increasing chromium content. [12].

The increased resistance to tempering 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 temperatures. 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 γ stabilizer. This gives beneficial effect on the mechanical properties of steels.

Nickel is generally added to martensitic steel castings to improve strength, ductility and corrosion resistance. For example, Souresny and Saver [11] 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 fatigue 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 functions of Mo as alloying element in stainless steels are : -

I. Imporvement 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 to tempering of martensitic stainless steels.

The martensitic stainless are employed in applications 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 components such as turbine blades. Molybdenum additions increases the maximum permissible operating temperatures for such parts [2].

2.4.4 Effect of Aluminium

Aluminium also forms intermetallic compound & is used to manufacture steels that can be age hardened by the approriate heat treatment.

2.4.5 Effect of other Elements :-

Mn- is also an austenite stabilizer, Mn is a possible substitute for Ni, which is both expensive & from time to time in short supply.

S, P & Se are added to 18/8 steel to improve their machinability as they have an embrittlening effects. P can also produce age hardening effects in austenitic steel. Si is added to resist scaling at elevated temperatures.

2.5 CRITERIA FOR THE 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 .Selection of a 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 characteristic 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 determined by the standard weight loss method [7].

General corrosion is often much less serious than the localized forms such as stress corrosion cracking, crevice corrosion in tight spaces or under deposits, pitting attack and inter granular attack in sensitized material such as various heat affected zones (HAZ) in fabricated parts, such localized corrosion can cause unexpected and 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 composition of the corroding medium including impurities.
- Physical state of the medium-liquid, gaseous, solid or combinations there of.
- Temperature variation.
- Oxygen content of the medium.

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

Even a slight variation in one of the above factors may lead to substantial variation in the performance of the material for a particular application and that is why corrosion resistance is considered to be 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 temperature must also be considered. Thus a product for arctic service must have suitable properties at subzero temperatures even though steady state temperature 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 STEELS

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

Application in marine systems[4]

Austentic and martensitc stainless steels have been used in marine power plants as super heater tubing and turbines 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 martensitiic steels, type 410 may rust in 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 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 chemical industry. However, the major tonnage 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 many 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 transsportation system [4]

The 11% Cr Or type A404 is being used increasingly to replace mild steel in specialized applications. One such application is for self contained tanks for fire fighting apparatus. Because such equipments must be ready for use at any instant , the storage tanks are always filled with water. A409 grade steel provides adequate corrosion resistance and is fabricated readily . Other applications include the joints used in exhaust system i.e. where temperature and corrosion are modest which allows use of type A410 and A409 steels.

Applications in the food Industry [4]

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

Application 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 EXHAUST VALVE

Austenitic valve steel containing 0.5%C, 9% Mn, Si 0.35% , Nickel 4%, Cr. 21% and N around 0.50% has been adopted by the automobile industry. As mentioned earlier this is a precipitation harden able type of stainless steel . It may be processed by regulating the rolling schedule in such a way that the finishing tempreture is close to 1100 ^{0}C. It could then be control cooled to get the required value of precipitation hardening . Alternately it can be solution treated by quenching from1050 to 1100 ^{0}C. (to ensure that the carbides are dissolved) to be followed by ageing at 700 to 750 ^{0}C, for the required length of time. It has also been suggested that an alternative method for attainment of required properties (hardness ~ 310 –350 VHN) is to by producing a "banded structure"

2.7.1 Probable casues of failure

Valves in automobiles are subjected to elevated temperature & high loads. Among the types of failures encountered in valves are those caused by

1. Burning - that is rapid oxidation by hot gases in exhaust valves.
2. Thermal Fatigue - usually occurs as valve guttering, begins with crack initation at sharp corners. Fatigue at the junction of the stem and head usually takes place .
3. Stress valve corrosion, over stressing & combination of these factors.

If in addition to normal loading a bending load is imposed on a valve, the combined stresses may lead to a failure at the point of maximum temperatures & stress.

2.8 CONCLUDING REMARKS

After a brief introduction to stainless steels in Sec. 2.1 , they are classified and described in the Sec. 2.4 Martensitic grade stainless steels are discussed in more details 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. in Sec. 2.7 Outlines the observation made on the performance of an austenitic steel used for making exhaust value . The aforementioned information is useful in formulating the problem.

CHAPTER –3

FORMULATION OF THE PROBLEM

3.1 GENERAL

The 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 WHY PRODUCT APPRAISAL

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

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

(b) the material has the right properties.

Neither is evidence of original thinking or even careful 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 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 Testing and Materials, Society of automotive Engineers, API standards etc., is helpful , but information must be used judiciously and must be relevant to a particular application [9] .

3.3 MATERIALS SELLECTION

Factors affecting materials selection are, corrosion resistance, resistance to a 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 some instances, a standard readily available component may be much less costlier 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.

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 condition. 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 material configuration and fabrication process, evaluating each against previously established criteria and making changes accordingly [16].

3.4 WORK PLAN

The main objective of this (*product*) appraisal was to asses whether the experimental material , which is in a developing stage in the some of the steel plants, is meeting with the specification as described in the standard.(27)

3.4.1. Objectives

Having thus laid down the modalities of selection and 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 steel in high technological applications.

The main objective of this product appraisal was to assess whether the experimental material which is in a developing stage in some of the mini steel plants , is meeting the specifications as described in the standards. Appraisal has been carried our mainly in the heat treated condition and related to a particular application namely automobile exhaust valves.

3.4.2. Step Wise Plan

The work plan relating to the investigation is given below :

1. To eliminate the presence of solute seggregation observed in valve steal.
2. To revaluate the heat treating cycle employed to obtain the stipulated value of hardness namely (311-345 VHN).
3. To asses whether development of a banded structure is pre requisite to obtaining the final hardness
4. Whether using alternative heat treating cycle(s) is useful in eliminating the central segregation.
5. To asses whether the overall processing cycle needs to be altered to obtain a material that is free of segregation both in the semi-rolled conditions .
6. To optimize the microstructure so as to obtain the stipulated mechanical properties .

CHAPTER 4

EXPERIMENTAL TECHNIQUES AND PROCEDURES

4.1 MATERIAL

The material used for this study was austenitic steel. It was made available by M/S STARWIRE (INDIA) LIMITED, BALLABHGARH, (HARYANA) in the form of rectangular & round samples in semi - rolled /as rolled condition. The allowable compositional variations for the grade of steel is as shown in the table given below :-

ALLOYING ELEMENT	Cr	Ni	C	Mn	Si	S	P	Mo	Al
%	19.44 - 21.05	3.2 - .3.5	0.54 -0.58	8.96- 9.2	0.94- 2.156	0.01 max.	0.018 max.	0.04 max	0.0 04

4.2 HEAT TREATMENT

Heat treatment of semi rolled/ as rolled samples were carried out in muffle furnaces whose temperature was controlled to ± 5 ^{0}c .

The temperature suggested in the standard are 1150^{0}C, 1175 ^{0}C and 1200 ^{0}C. Soaking time was 1 hrs ,2 hrs , 3 hrs respectively for all the three temperature . In some instances the semi rolled/ as rolled samples were cut into two parts before heat treatment. One part was air cooled while the other part was water quenched after heat treatment was completed.

4.3 PREPARATION OF TEST SPECIMENS

For initial studies a total of 37 specimens were taken . In beginning 10 specimens were heat treated. In the second stage of work 18 specimens in the semi - rolled condition of rectangular shape (3cm x 5cm x 2cm) were studied . Later on 9 specimens in the as rolled conditions (of the size 1.5cm to 2.5 cm dia. and 1cm in length) were heat treated. During the final stages, property assessments were carried out on as-rolled specimens in the heat treated condition.

4.4 METALLOGRAPHIC STUDY

Optical microscope MeF-3 was employed for the metallographic studies. Microphotographs were taken at magnification of 50x, 100x, 200x, and 400x in etched and unetched condition. For the study of microstructure and grain size, specimens were etched with freshly prepared etchant, ferric chloride of composition, $FeCl_3$- (5 gms.) + HCl (50 ml) +H_2O(100 ml distilled water) +2 to 3 drops of HNO_3 . [19] after they had been polished by employing the standard procedure laid down for the purpose.

Macro etching was done, with 20% Con. HCl + 30%Con. HNO_3+50%H_2O solution for a time period of 10-15min to assess whether segregation occured.

4.5 Hardness Testing

This involved assessment of hardness in the heat treated condition. Hardness was measured on Vicker's hardness testing machine at a load of 30Kg. A min. of 10 impressions were taken on each specimen at various locations & average of these values, along with standard devitation found out.

4.6 Tensile Properties

Tensile tests were conducted on samples made as per the details(fig. 4.1)using HOUNSFIELD TENSOMETER with a provision to vary the cross head speed & values of the ultimate tensile strength (UTS), proof strength,(PS), Elongation (% EL) and % Reduction in Area (% RA) were calculated and recorded.

4.7 EPMA

Electron probe micro-analysis was carried out on the JEOL, JXA - 8600 M Electron probe micro analyser. The studies proved helpful in assessing the level of segregation and in finding out the composition of inclusions.

4.8 X-RD ANALYSIS

The specimens were subjected to structural investigations on a PHILLIPS diffractometer PW 1140/90, employing an iron target and a Mangenese filter, at a voltage of 35 KV and a current of 12 mA.

Specimens were polished and lightly etched, were scanned from 50 to 100^0, time constant and scanning speeds were kept at 2 seconds and 1^0 per minute.

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CHAPTER 5

RESULTS AND DISCUSSION

5.1 GENERAL

In this Chapter the data generated, relevant to carrying out product appraisal, has been reported. It comprises the effect of heat treatment on the hardness and microstructure pertaining to semi-rolled and as-rolled material. Subsequently, data pertaining to mechanical properties of the as-rolled material in the heat treated condition has been collated and reported. Inclusion rating has been carried out to report upon the cleanliness of steel. EMPA data on level of segregation has also been included. Some results dealing with high temperature tensile properties have also been included to carry out an indepth analysis of the usefulness of the product for the intended application.

5.2 RESULTS

5.2.1Chemical Analysis

Instrumental analysis of the experimental material is reported in table 5.1. It emerged that the analysis is in conformity with that specified in the relevant standards.

5.2.2 Effect of Heat Treatment on Hardness

The initial characterization of the material was carried out by subjecting the semi-rolled and as-rolled material to the stipulated heat treatment cycle(s), followed by finding out the hardness. The resulting data is summarized in the tables 5.2 to 5.4.

From an analysis of the data thus reported, it emerged that :

1. For the semi-rolled air cooled condition, hardness initially increased on increasing the soaking period from 1 to 2 hours and there after decreased slightly on raising the soaking period to 3 hours (Heat treating temperature 1150^0C) (Table 5.2).
2. On raising the temperature to 1175^0C, the hardness initially increased, on increasing the soaking period from 1 to 2 hours. A further increase in it to 3 hours had little effect on hardness (Table 5.2).
3. On heat treating from 1200^0C, hardness was more or less independent of the soaking time (Table 5.2).
4. On subjecting the semi-rolled material to water quenching, the variation in hardness with time was a function of the heat treating temperature and time. On quenching from 1150^0C, the hardness initially increased on raising the soaking period from 1 to 2 hours and thereafter declined on raising the period to 3 hrs (Table 5.3).
5. On raising the temperature to 1175^0C, the hardness decreased with soaking period. A similar trend was observed on water quenching from 1200^0C (Table 5.3).
6. The overall level of hardness on water quenching was lower than the one obtained on air cooling the semi rolled specimens (Table 5.2 and 5.3).
7. Considering the hardness data on the as rolled material in the water quenched condition, (Table 5.4), it emerged that
    - (i) On heat treating from 1150^0C, the hardness initially decreased on raising the soaking period from 1 to 2 hrs and there after was nearly a constant.
    - (ii) On heat treating from 1175^0C there was a decrease in hardness as the soaking period was increased from 1 to 3 hrs.

(iii) On raising the soaking temperature to 1200^0C, the hardness was independent of the soaking period.

5.2.3 Effect of Heat Treatment on Microstructure

The as-received material in the semi-rolled and as-rolled condition comprised a two phase microstructure. The samples were non-magnetic and hence denote the material/microstructure to be austenite based. Looking to the composition in table 5.1, the second phase would be a combination of carbides and nitrides.

Semi-Rolled Specimens

(i) On water quenching from 1150^0C, after soaking for 1 hr the microstructure comprised two phases as above (Fig. 5.1(a)). The microstructure was banded and the second phase was in the form of very small lamallae/spheroids (Fig. 5.1(b)).

(ii) On increasing the soaking period to 3 hours, the general features were as before except that some coarsening was observed (Fig.5.1(c)).

(iii) On heat treating from 1175^0C, after soaking for 1 hr, the general features observed were similar to the ones observed on heat treating from 1150^0C (Fig. 5.2(a)). The layered nature of the second phase is in evidence. At the grain boundaries, chain like formation of the 'second phase' is in evidence (Fig.5.2(b)).

On increasing the soaking period to 3 hrs the matrix comprised well defined grains with twins (Fig.5.2(c)). The second phase was in the form of dark globules, which were also located along the grain boundaries (Fig.5.2(c)). This aspect is more clearly marked in (Fig.5.2(d)), where the second phase particles were located both along the grain boundaries as well as at the triple points.

(iv) On heat treating from 1200^{0}C, (1 hr soaking period) the structure is similar to that observed on heat treating from 1175^{0}C (3 hr soaking period), except for the grain size which is coarser (Fig. 5.3 (a & b)).

On soaking for 2 hours, the matrix as before comprised grains with twins and the second phase was in the form coarse-dark globules (Fig. 5.3 (c) and (d)). The second phase observed resembled as though non-metallic inclusions were present atleast in some of the locations (Fig. 5.3(d)).

On increasing the soaking period to 3 hours, the microstructure (Fig. 5.3(e)) was similar to the one observed on soaking for 2 hours (Fig.5.3(c)).

(v) Representative micrographs of the semi-rolled specimens in the air cooled conditions have also been included for the purpose of comparison (Fig. 5.4(a) & (b)).

At 1150^{0}C, 1 hour soaking period the microstructure was banded. The amount of second phase was more than what is obtained in the as quenched condition (Fig. 5.4(a)) & (Fig. 5.1(a)). On raising the soaking period to 3 hours the microstructure was similar to that in (Fig. 5.4(a)) and hence has not been included.

At 1200^{0}C 3 hours soaking period the matrix comprised coarse grains and the amount of the second phase was reduced. However banding was seen to present (Fig. 5.4(b)).

As-Rolled Specimens

(vi) On water quenching from 1150^{0}C, after soaking for 1 hr, the microstructure is not well defined at low magnification. The grain size was small and banding if any was at a minimum (Fig. 5.5(a)). On raising the magnification grains were visible and presence of second phase was clearly indicated (Fig. 5.5(b)).

On raising the soaking period to 2 hours, the two phase nature of microstructure becomes clear even at lower magnification (Fig. 5.5(c)). Higher magnification micrograph did not reveal any thing specific except for some coarsening (Fig.5.5(d)).

On raising the soaking period to 3 hours, the nature of microstructure is more clearly defined. The second phase particles, which are both dark and globular and occassionaly forming bead like chains, are clearly visible (Fig. 5.5 (e) & (f)).

(vii) On heat treating from 1175^{0}C, soaking for 1 hr microstructure is similar to the one observed at 1150^{0}C 1 hours Fig. 5.6(a) & Fig. 5.5(a). At higher magnification it was similar to that obtained at 1150^{0}C 3 hours (Fig. 5.6(b) & Fig. 5.5(f)).

On raising the soaking period to 2 hours, the coarser γ-grains Fig. 5.6(c) resembled those observed is Fig.5.5(c). At higher magnification (Fig. 5.6(d)) the situation was similar (Fig.5.5(d)).

On raising the soaking period to 3 hours the structure was similar to the one obtained by heat treating from 1150^{0}C, 3 hours, except that the overall coarsening was more [Fig. 5.6(e) & (f)) and (Fig. 5.5(e) & (f)).

(viii) On heat treating from 1200^{0}C, soaking for 1 hour, the microstructure comprised grains with twins and a second phase in the form of dark globules (Fig. 5.7(a)). These globules resolved themselves in the form of a second phase showing some layered structure Fig. 5.7(b).

On raising the soaking period to 3 hours the matrix revealed the presence of grains, twinning was more marked Fig. 5.7(c). At higher magnification the second phase was observed to be present inside the grains as well as at the grain boundaries in the form of beads and spheres Fig. 5.7(d).

5.2.4 Effect of heat treatment on the tensile behaviour

The effect of solutionizing temperature and time followed by ageing at different temperatures (730, 750 and 780^0C) for different ageing periods (6, 10, 14, 16 hrs) on the tensile behaviour is summarised in table 5.5 It is seen that 0.2% proof stress varied from 590 to 866 MPa, UTS from 910 to 1280 MPa, % elongation from 4.9 to 20.98 %, % reduction in area from ~ 10 to 12% and hardness from 29 to 37 Rc.

A closed scrutiny of the data revealed specific trends which are given below:

(i) For solutionizing temperature of 1120^0C and aging time of 6 hours increasing the ageing temperature from 730 to 780^0C, led to a decrease in UTS and 0.2% P.S. For aging temperature of 780^0C, increasing the ageing time from 6 to 10 hours lead an improvement is 0.2% P.S. as well as U.T.S. with % elongation values being quite favourable.

(ii) For solutionizing temperature of 1150^0C and ageing time of 6 hours, increasing the ageing temperature from 730 to 780^0C led to a decrease in P.S. where as the decrease in the U.T.S. was very small. The overall elongation values could be considered as reasonable.

For ageing temperature of 780^0C, increasing the ageing time from 6 to 10 hours led to an increase in P.S. as well as U.T.S., whereas the % elongation values were found to be reasonably. And further increasing the ageing time to 14 hours led to a decrease in P.S. as well as U.T.S. with the elongation values being very good (~ 15 to 19%).

(iii) On increasing the solutionizing temperature to 1180^0C and at an ageing time of 6 hours increasing the ageing temperature from 730 to 750^0C led to an increase in P.S. as well as U.T.S. with elongation values being reasonable. On increasing the temperature to 780^0C, there was a decrease in P.S. as well as U.T.S. Further increasing the ageing time to 10 and 14 hours led to a decrease in P.S. while U.T.S. was more or less unchanged except on soaking for 14 hours where infact it was higher. Percent elongation values were consistently good and in the range of ~ 11 to 20 %.

(iv) For the same ageing temperature of 750°C and the ageing time of 16 hours, increasing the solutionizing temperature from 1120 to 1180°C led to a continuous decrease in P.S. and U.T.S. with % elongation values being reasonably good except for the last heat treatment for which the solutionizing temperature was 1180°C.

5.2.5. X-RD Analysis of As-Rolled Specimens

Selected as-rolled specimens, in the heat treated condition were subjected to x-ray diffractrometric analysis with a view to find out the nature of the second phase present. The data thus obtained is summarised in the Tables 5.6 to 5.10. The following inferences may be drawn based on the analysis carried out.

5.2.5.1 Carbides/Intermetallic Compounds (Tables 5.6 to 5.9)

(i) On heat treating from 1150°C (1 hr, WQ), the carbides present were mostly M_5C_2 and Mr_7C_3 type. Interestingly, rather than mixed carbides being present, the indexing carried out suggested the likely presence of straight carbides. Additionally possible formation of $Mn_{15}C_4$ type of compound was also indicated.

(ii) On raising the soaking period to 3 hours (1150°C, WQ) the nature of diffractogram was nearly similar.

(iii) On raising the temperature to 1175°C (3 hr, WQ) the second phase now mostly comprised either $(Cr, Fe)_7C_3$ type or more specifically a mixed carbide of the type $Cr_7C_3+Mn_7C_3$.

(iv) On heat treating from 1200^0C (1 hr and 3 hr, WQ) a marked tendancy towards possible formation of Fe_5C_2/Mn_5C_2 and Fe_7C_3/Cr_7C_3 was revealed. Similarly the possible existence of $Mn_{15}C_4$ was also indicated.

5.2.5.2 Nitrides

Table 5.10 reveals that in the representative heat treated specimens the possible occurance of Fe-Nitride and Al-Nitride is indicated.

5.2.6 Inclusion rating/Analysis

Data pertaining to the analysis of inclusion rating carried out optically is summarised in table 5.11 and shown in Fig. 5.8. It was observed that mostly A, B and D type of inclusions were present that too belonging to the thin series.

EPM analysis of a typical field of view is shown in table 5.12.

5.2.7 Data on elemental segregation

A representative as-rolled specimen of the experimental material was polished in the longitudinal direction and observed under EPMA to find out the level of segregation if present. The scanning was done from one end of the sample to the other across the dia. Typical results are summarized in the tables 5.13 to 5.15. The variation in the element concentration could be noticed.

5.3 DISCUSSION

5.3.1 Hardness

In order to arrive at a detailed appraised of the behaviour of the experimental steel, it is necessary to understand the transformation behaviour of the material. The first indication in this regard is that it is an austenite based steel which is strengthened through precipitation hardening. Looking to the composition of the material (Table 5.1) it is not difficult to appreciate that the second phase will mostly comprise a combination of carbides and nitrides. Since the extent of precipitation will be a function of the temperature and time, these two parameters together with the cooling rate will play an important part in deciding the properties most likely to be attained.

Considering the transformation behaviour of the semi-rolled material, on air cooling from 1150^{0}C (soaking time 1 hr), what ever amount of the second phase is dissolved in austenite is most likely to precipitate out during air cooling, resulting in a hardness of ~ 370 HV_{30} (Table 5.2). On raising the period to 2 hours, some more of the second phase will dissolve most of which will reprecipitate on air cooling leading to a slightly higher hardness of ~ 390 HV_{30}. By the same token of argument a still higher hardness should have been attained on increasing the soaking period to 3 hours. However at this soaking period the level of coarsening will be higher than what is observed at 2 hour soaking period, resulting in a slightly lower hardness (table 5.2).

That the above analysis is correct is duely borne out by examining the hardness data on heat treating from 1175^{0}C, wherein the hardness increased on raising the soaking period from 1 to 2 hours and thereafter it remained more or less unchanged on increasing the soaking period to 3 hours.

On raising the heat treating temperature to 1200^{0}C, although a larger amount of second phase will dissolve into austenite with an increase in soaking period, none the less the extent of coarsening of the reprecipitated second phase will be more leading to a level of hardness which is lower than what is obtained on heat treating from 1175^{0}C (Table 5.2).

Considering now the hardness values obtained in the semi-rolled specimens on water quenching, a first level general observation is that the overall level of hardness as influenced by heat treating temperature and time was lesser than what is obtained on air-cooling (Table 5.3). This is because on water quenching we are effectively preventing atleast partly the reprecipitation of the second phase during cooling. Accordingly we should got relatively lower overall hardness values on heat treating from 1200^0C in comparison to the hardness values obtained on heat treating either from 1150^0C or 1175^0C. This is what has been observed. As is indicated in (Table 5.2), the optimum heat treating temperature for obtaining a higher level of hardness is once again 1175^0C (Table 5.3).

The hardness values summarised in Table 5.4 which pertain to the effect of heat treating parameters in relation to the as-rolled condition, can be explained on the basis of a similar reasoning put forward above.

5.3.2 Microstructure

A detailed account of how the microstructure is influenced by temperature and time of soaking and the cooling rate has been given in the section 5.2.3. The basis on which the formation of different microstructures can be explained has been outlined in the initial paragraph of section 5.3.1. It would therefore be possible to correlate the changes in the microstructure as summarised in the figure 5.1 to 5.7 with the hardness values summarised in table 5.2 to 5.4. Certain broad guidelines to interprete the microstructure and correlating it with hardness are as follows :

(i) An increase in temperature from 1150^0C to 1200^0C for a given soaking period will accelerate the tendency to form austenite. This tendency will be more marked on quenching and relatively lesser marked on air cooling.

(ii) For a given heat treating temperature the austenite forming tendency will be higher at a higher soaking period especially if quenching is employed.

(iii) The volume fraction of the second phase will decrease with an increase in heat treating temperature (for a specified period).

(iv) A similar situation will exist if the soaking period is increased at a specified heat treating temperature.

(v) A decrease in the volume fraction of the second phase (due to coarsening) will lead to a decrease in hardness.

The aforementioned analysis satisfactorily explains the microstructures observed and so also the inter-relation between microstructure and hardness.

Having thus explained structure-related observations an effort will now be made to comment upon specific features.

(i) The presence of banding and its persistence on heat treating from higher temperatures (Fig.5.1(a), Fig.5.2(a), Fig.5.4(a) & (b), needs to be explained. It may be mentioned that banding is perhaps a feature associated with FCC structure. Its occurrence is perhaps correlated with the accommodation of localised stresses and thus its formation may be a precurser to the formation of twins (24).

(ii) It is not clear if the occurence of banding is a necessary condition for the formation of the desired microstructure, more so because banding is not desirable from the point of view of mechanical properties.

(iii) Even after soaking for 3 hours at a temperature of 1200^0C the second phase is not fully dissolved. Infact the second phase is of two distinct varieties as shown in the Fig. 5.7.

(iv) Occurence of twinning in the austenitic based matrix, as extensively found (Fig.5.2(c), Fig.5.3(c), Fig.5.3(e), Fig.5.4(b), Fig.5.7(a) & (c)), is a feature normally associated with FCC structure, and therefore the observation stands explained.

(v) The second phase observed on heat treating from 1200^0C at 1 hour soaking period (Fig. 5.7(b)) could be either a nitride and or an intermetallic compound.

(vi) On prolonged soaking at 1200^{0}C, the formation of the second phase along the grain boundaries and its nature is not clearly understood and needs further investigation (Fig. 5.7(b) and (d)).

5.3.3 X-RD Analysis

X-RD analysis is being considered before commenting upon the mechanical properties as it would help in arriving at a better understanding of the correlation of the mechanical properties with the structure. There are certain noteworthy features such as (i) near absence of mixed carbides (ii) presence of Cr_7C_3 + Mn_7C_3 and/or Fe_5C_2 and Mn_5C_2, (iii) likely presence of $Mn_{15}C_4$ and (iv) existance of nitrides more or less independent of heat treating temperature and time (Tables 5.6 to 5.10). These observations agree well with the XRD based analysis arrived at while designing special cast irons for marine applications (25).

These observations compliment the microstructural features already discussed in the section 5.3.2 e.g. the specimens corresponding to the heat treatment 1200^{0}C 3 hr WQ is most likely to contain a nitride in addition to Fe_5C_2 and Cr_7C_3 (25).

5.3.4 Mechanical Properties

The room temperature mechanical properties summarized in the Table 5.5 can be considered as reasonable and well within the limits of acceptance as laid down in the standards. A careful persual of the data contained in Table 5.5 shows certain systematic trends e.g. for the solutionzing temperature of 1120^{0}C and 1150^{0}C (ageing time 6 hrs) a decrease in the UTS/PS with an increase in the ageing temperature is due to a reduction in the volume fraction of the 2nd phase/its coarsening. Photomicrographs corresponding to the solutionsizing/ageing heat treatments have not been included to avoid repetition.

Similarly, a very graudual decrease in the PS with practically no change in the UTS on increasing the ageing time from 6 to 14 hrs at an ageing temperature of 780^0C and solutionizing temperature of 1180^0C indicates that there is little change in the precipitation kinetics.

Barring a few instances the % elongation values at room temperature are reasonably good and reveal not only the soundness/usefulness of the microstructure but also that the material (i) is reasonably 'clean' in terms of the presence of inclusions/impurities and (ii) 'homogeneous' (segregation within allowable limits). This becomes further evident on considering these two aspects.

5.3.5 Inclusion Rating

Mostly two types of inclusion have been seen to be present namely type 'A' sulphides and type 'D' (oxides) with occasional presence of type 'B' alumina that too belonging to the thin series (Table 5.11 (a) & (b)). Silicates have been found to be absent, which is a desirable feature since automible manufacturers using this material as valve steel insist upon the absence of silicates (26). This observation augers well for the steel making practice adopted. EPM anlaysis of the inclusions present (Table 5.12) further confirms the above.

5.3.6 Elemental Segregation

Representative elemental segregation based data, as found out thrugh using EPMA, (Tables 5.13-5.15) reveals that the amount of the elements is a little higher closer to the periphery and reduced as the central portion is approached. By all accounts the elemental variation is reasonable especially looking to the multiplicity of the elements present and the high alloy content of the material.

5.3.7 Over View

The aforementioned discussion reveals that the experimental material has been investigated in fair detail and the mechanical properties it exhibits can be correlated with the microstructure. The overall material is sound for the intended application and this is duely justified based on the analysis of inclusions/level of segregation. It exhibits properties which are comparable with those indicated in the standard specifications. It is therefore expected to also fulfil the requirments laid down in relation to the high temperature properties which should be as given below (27) :

High temperature properties in solutionsed and aged condition

Temperature	500	550	600	650
Tensile strength MPa	645	595	550	490
0.2% proof stress, Mpa	355	320	295	265

CREEP Rupture Strength, MPa

Hours	**650^{0}C**
1,000	185

CHAPTER 6

CONCLUSIONS AND SUGGESTIONS FOR FUTURE WORK

6.1 CONCLUSIONS

Under the existing experimental conditions the following conclusions are arrived at:

(i) Hardness is a function of heat treating temperature, time and cooling rate.

(ii) The desired level of hardness i.e. ~ 30-32 Rc can be arrived at through a number of heat treating cycles.

(iii) The microstructure of the material consists of austenite matrix and dispersed second phase namely carbides and nitrides.

(iv) The carbides mostly comprised Fe_5C_2/Mn_5C_2/Cr_7C_3/Mn_7C_3 and $Mn_{15}C_4$. Possible existance of Fe_7C_3 has also been revealed. The carbide(s) to be present is a function of heat treating temperature and time.

(v) Besides the above, the presence of Fe-nitride and Al-nitride is revealed.

(vi) The material behaved satisfactorily from the point of view of inclusions and elemental segregation point of view.

(vii) The tensile properties stipulated in the standards (27) namely Hardness ~ 32 HRc, 0.2% P.S. ~ 580 MPa, Tensile strength ~ 950-1200 MPa, % Elongation ~ 8, % R.A. ~ 10 could be obtained through various combinations of solutionizing and ageing treatments. The optimum among the same is 1150^{0}C solutionizing temperature, agenging temperature of 780^{0}C, ageing time 10 hrs corresponding to which 0.2% P.S. is 866 MPa, U.T.S.=1280 MPa, % RA=11.5, HRc=35 and % elongation ≈ 15 to 20%. A number of other heat treatment cycles can also be suggested.

6.2 SUGGESTIONS FOR FUTURE WORK

The following suggestion are indicated to make the appraisal more comprehensive :

(a) A detailed investigation into the structural changes by X-RD.

(b) Estimation of the high temperature tensile properties.

(c) Estimation of creep rupture strength.

(d) High temperature oxidation studies.

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BS - 349 S 54, SAE - EV8, and DIN - 1.4871

Figures
&
Tables

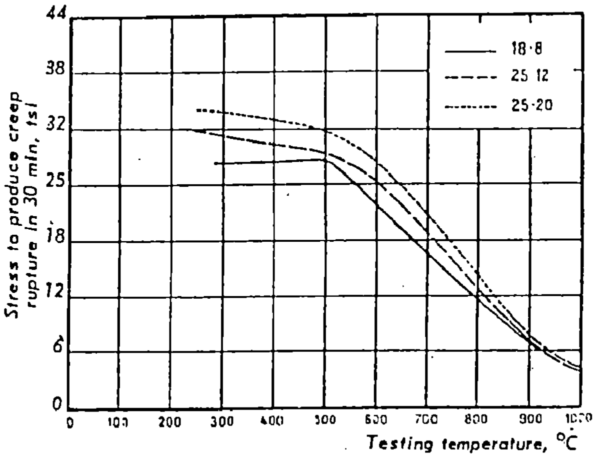
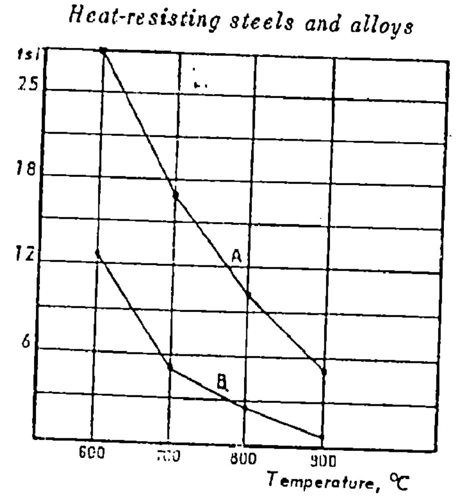


Fig.2.1 Creep-rupture stresses for a 30-minute life at different temperatures, on different austenitic steels (11)



A - austenitic steel with 0.4% C, 13% Cr, 13% Ni, 2% W;
B - Si-Cr steel with 0.4% C, 8.5% Cr, 3.0% Si

Fig.2.2 High temperature creep-rupture tests on an austenitic and a martensitic valve steel (11)

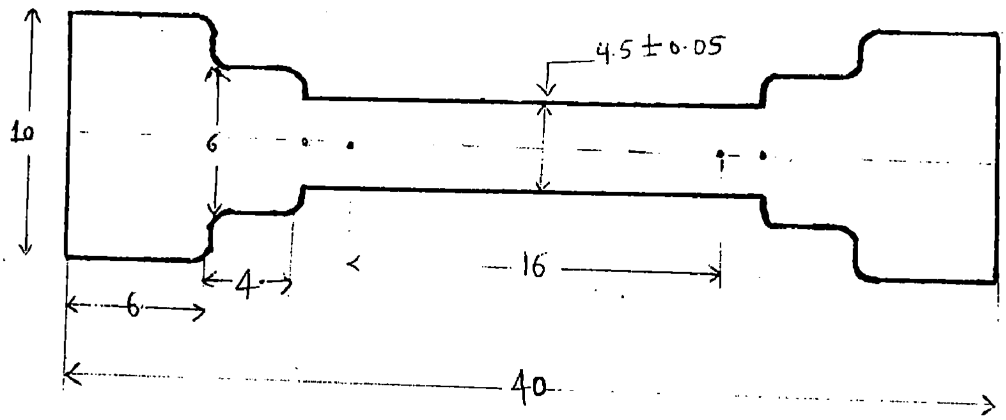
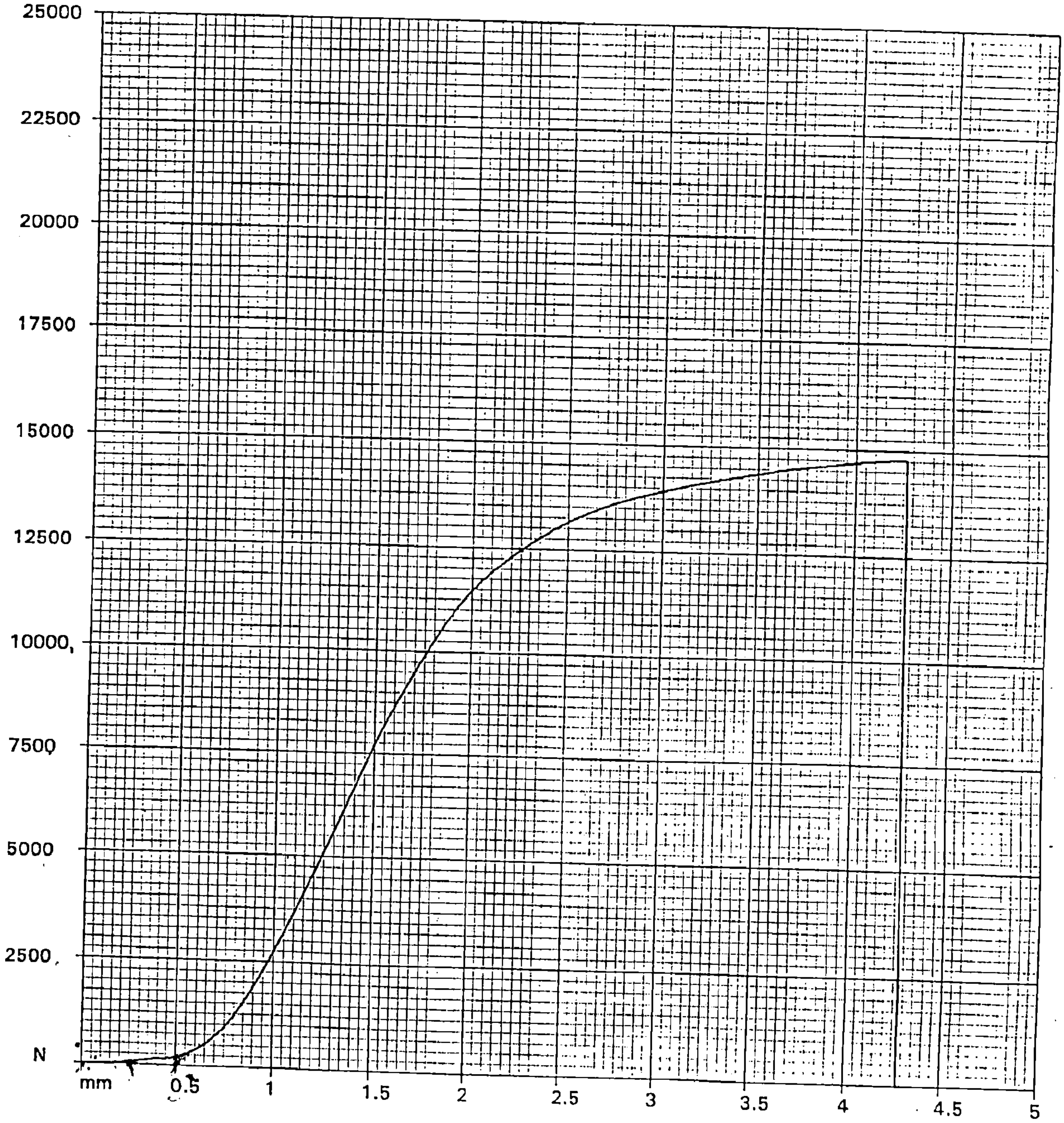
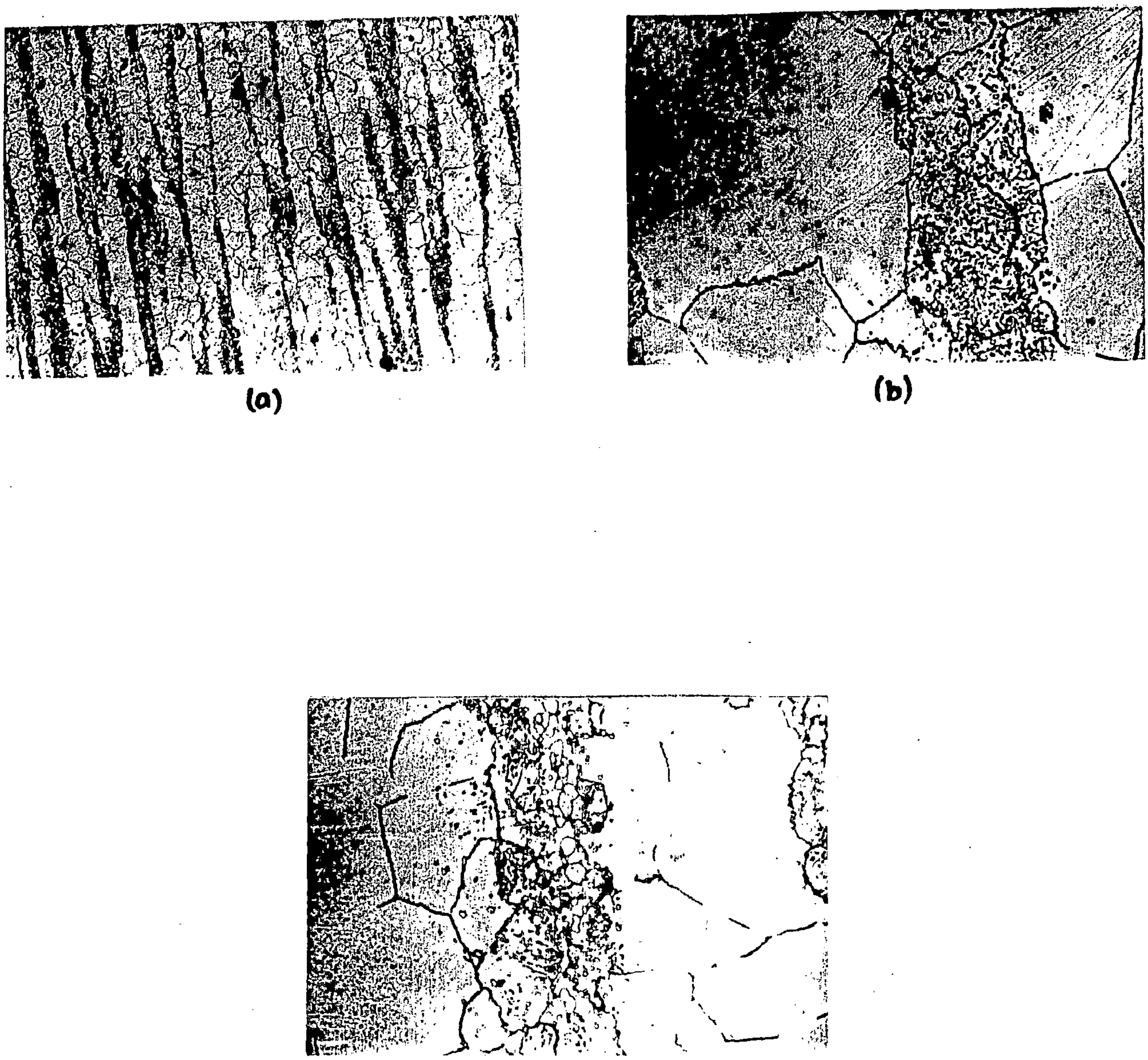


Fig. 4.1 Tensile specimen (all dimensions in mm)

Fig. 4.2 Hounsfield test report (soln. temperature 1120^0C, ageing temperature 780^0C for 6 hrs)



MICROSTRUCTURE OF SEMI-ROLLED SPECIMENS



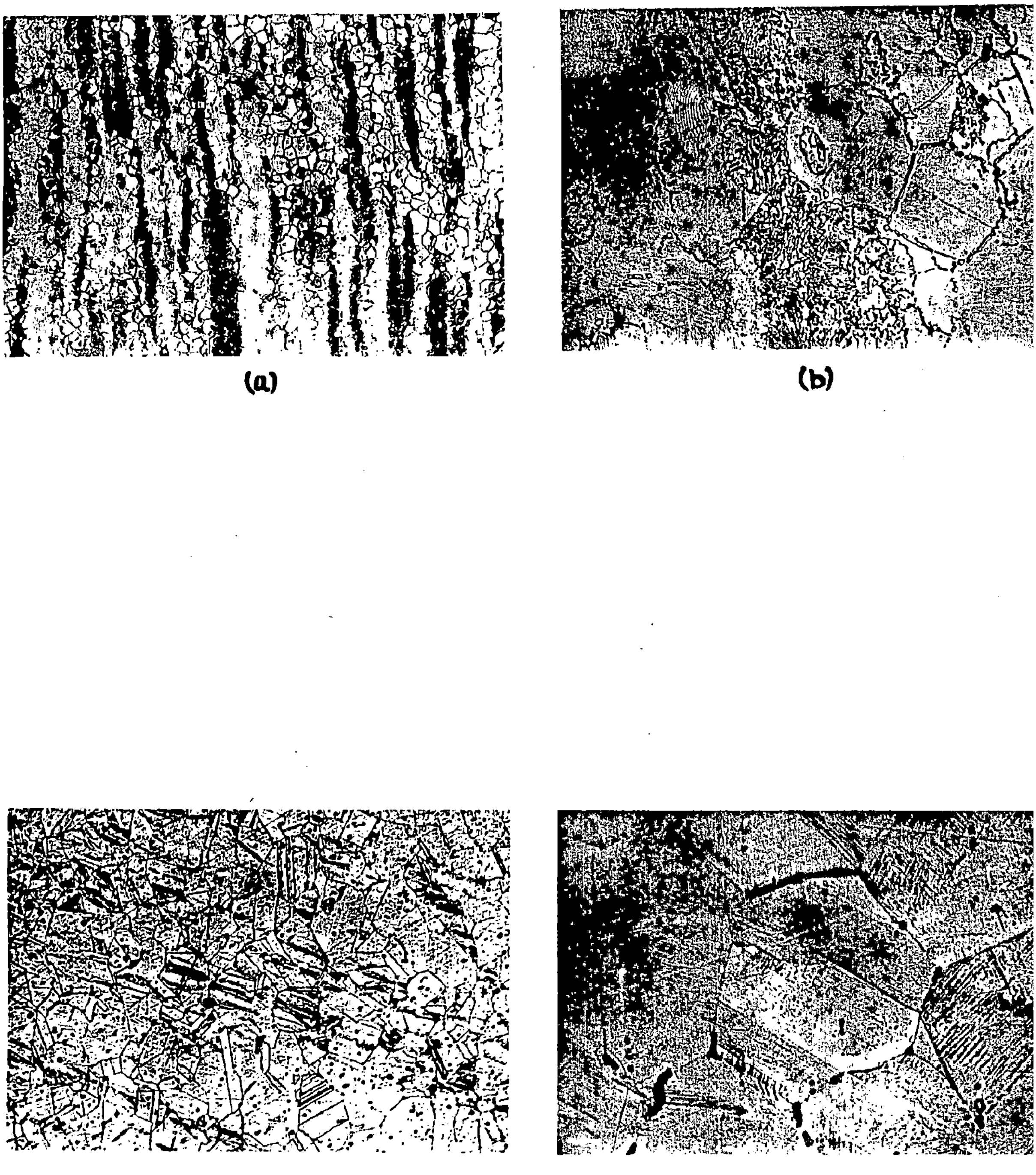
(a)

(b)

(c)

Fig. 5.1 : Temperature 1150°C

(a) 50X , (b) 400X 1hr. W.Q
(c) 3hr W.Q 400 X



(a) (b) (c) (d)

Fig. 52 : Temperature 1175 °C
(a) 50 X , (b) 400X 1hr W.Q
(c) 50 X (d) 400X 2hr W.Q

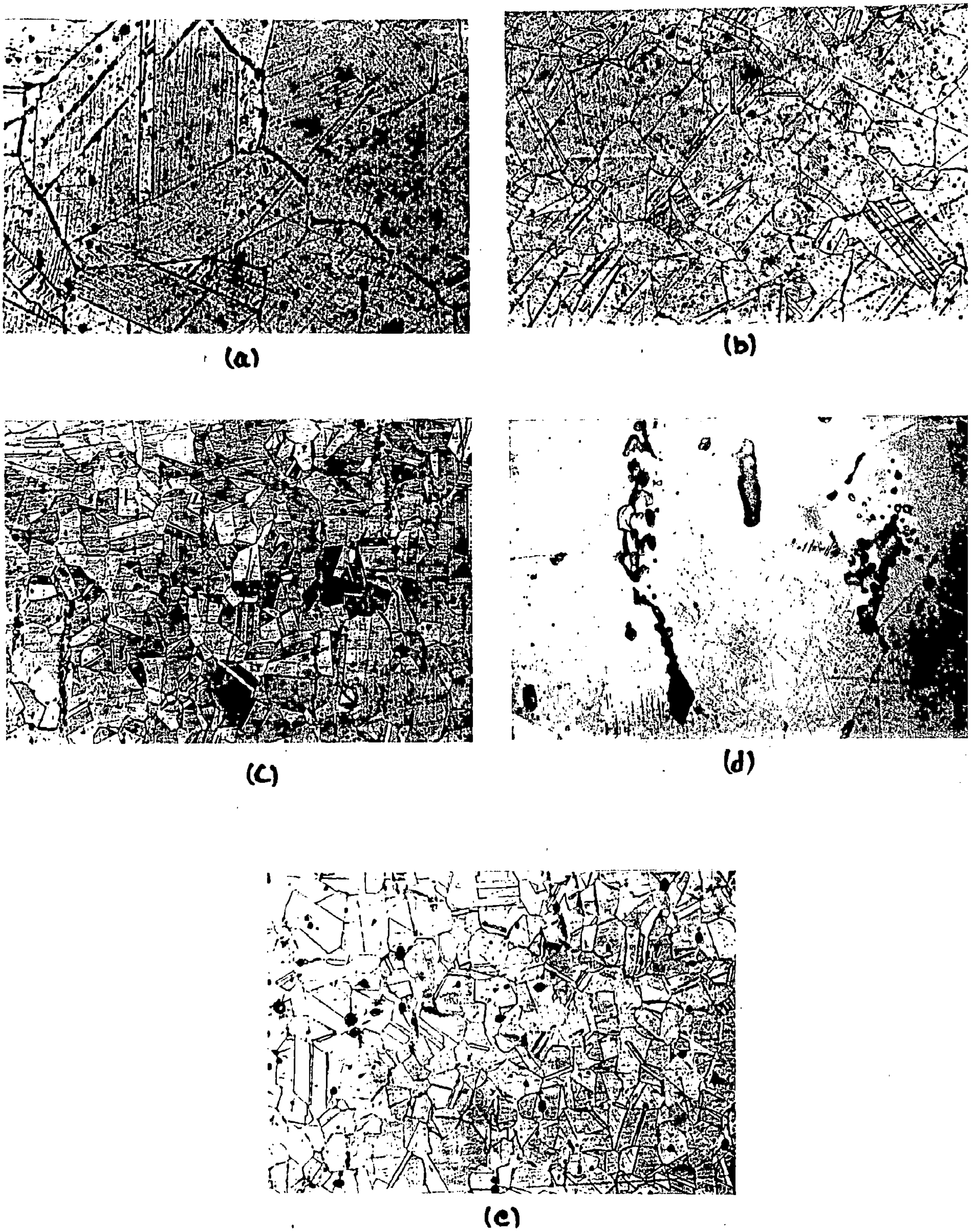

(a) (b) (c) (d) (e)

Fig. 5·3 : Temperature 1200°C

(a) 100 X 1hr W.Q. (b) 200 X 1 hr W.Q.
(c) 50 x 2hr W.Q. (d) 400 X 2hr W.Q.
(e) 50 X 3hr W Q

MICROSTRUCTURE OF SEMI-ROLLED AIR-COOLED SAMPLES

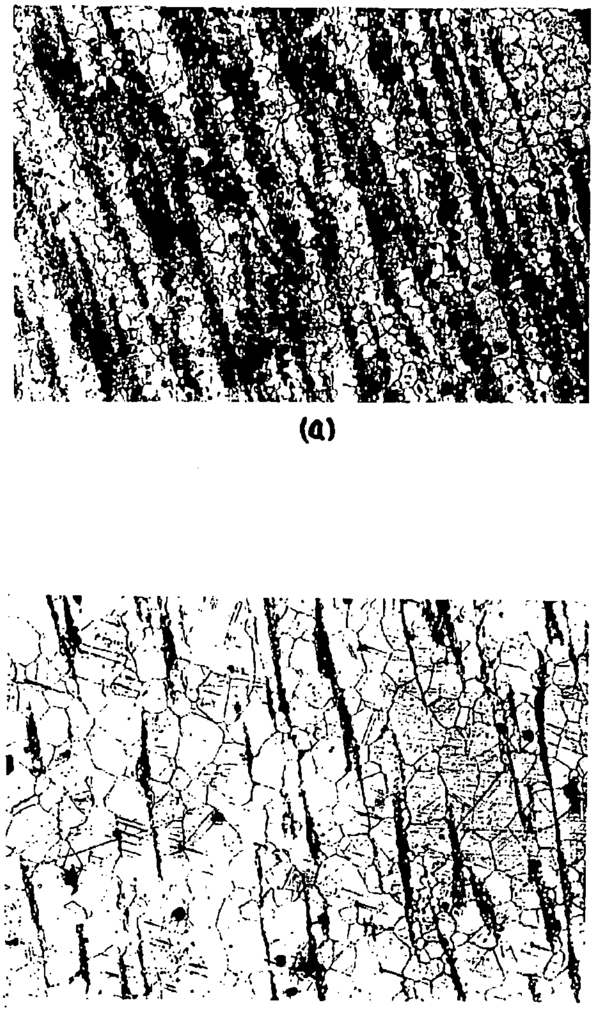
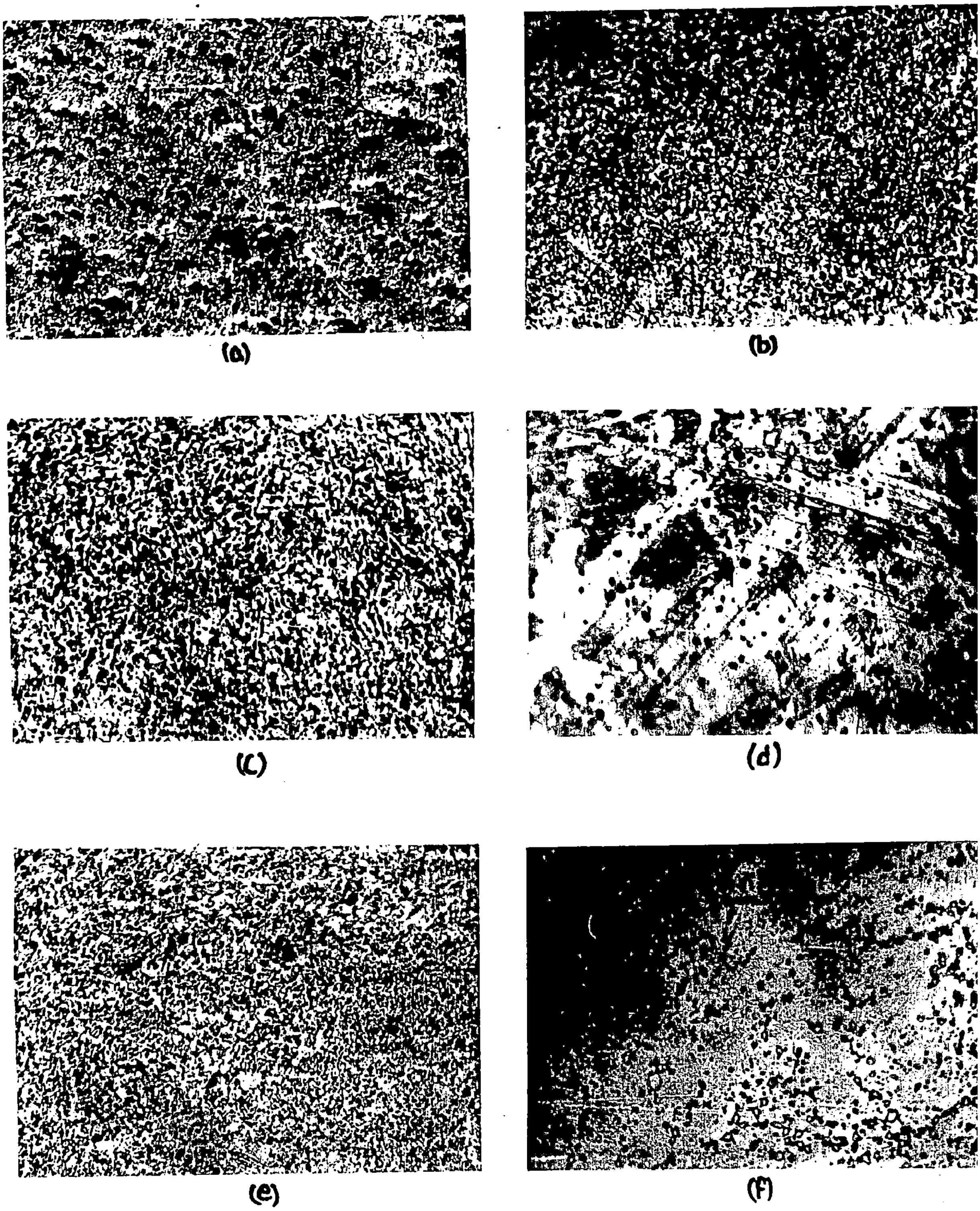


Fig. 5·4 : (a) 50x, 1150°C, 1hr. A·C
(b) 50x, 1200°C, 3hr. A.C

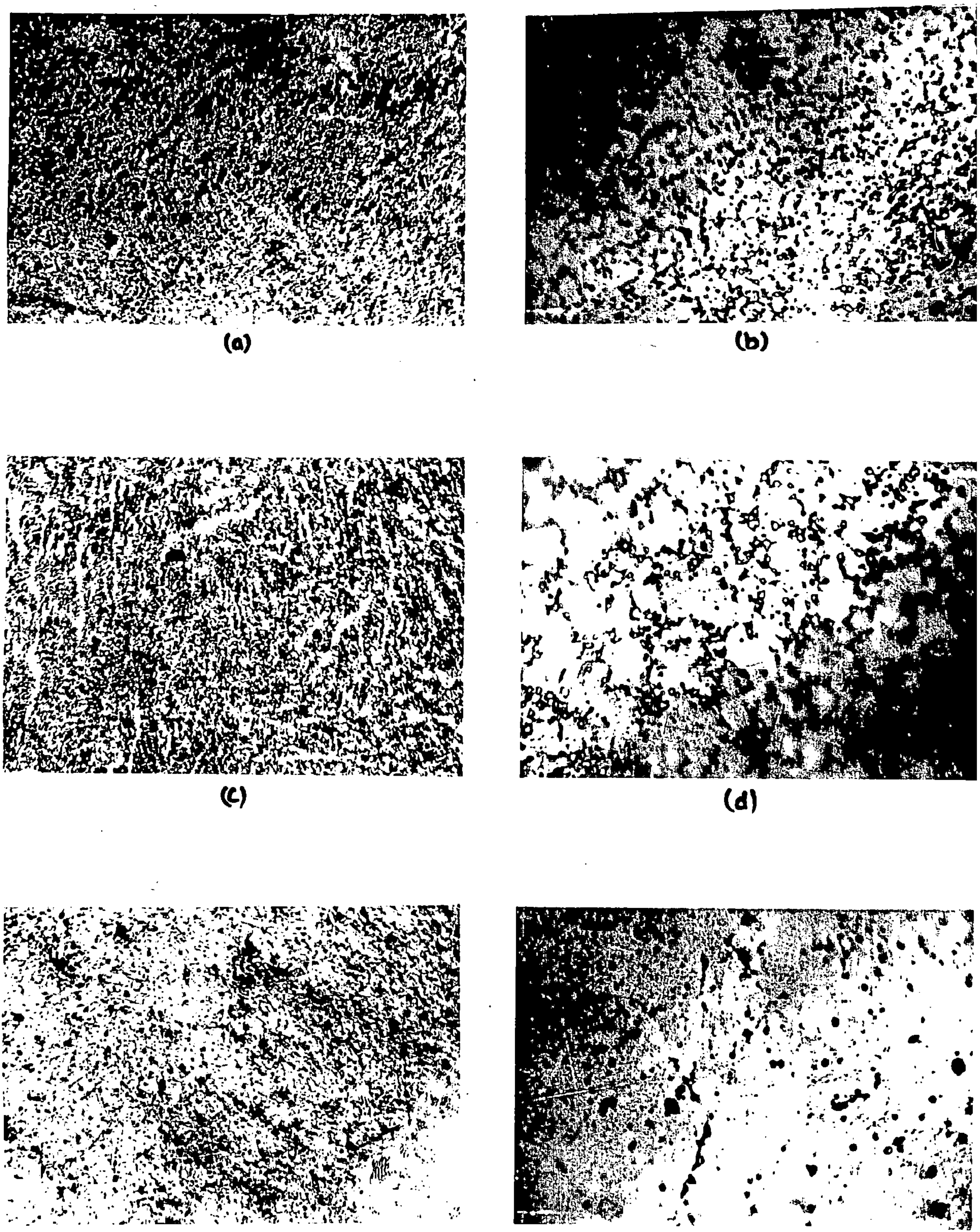
MICROSTRUCTURE OF AS-ROLLED SPECIMENS



(a) (b) (c) (d) (e) (f)

Fig. 5.5 : Temperature 1150 °C

(a) 50 x , (b) 200 x , 1hr . W.Q
(c) 50 x , (d) 400 x , 2 hr, W.Q
(e) 50 x , (f) 400 x , 3hr . W.Q



(a) (b)

(c) (d)

(e) (f)

Fig. 5·6 : Temperature 1175°C

(a) 50 x (b) 400 X , 1hr . W.Q
(c) 50 x (d) 400 X ; 2hr . W.Q
(e) 50 x (f) 400 X , 3hr . W.Q

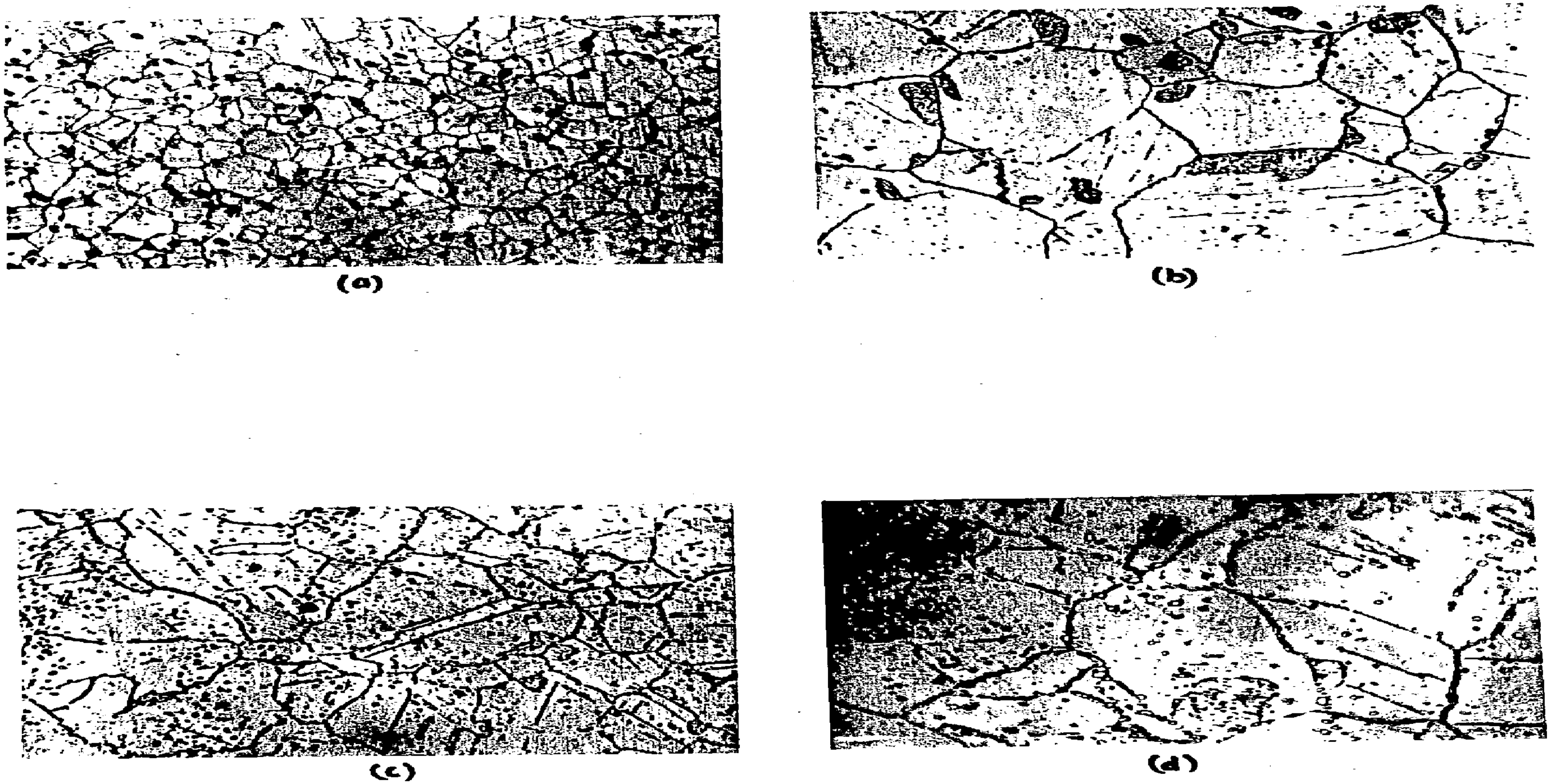
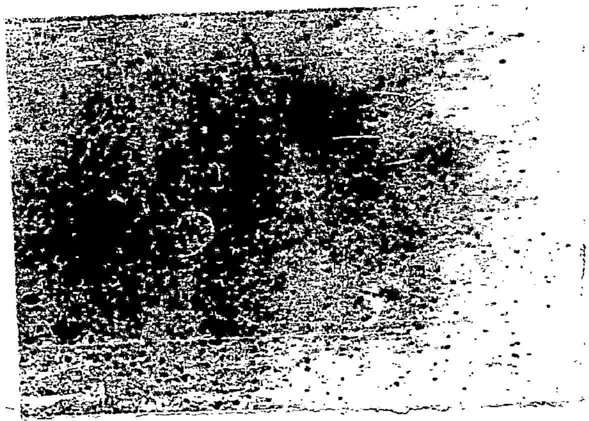
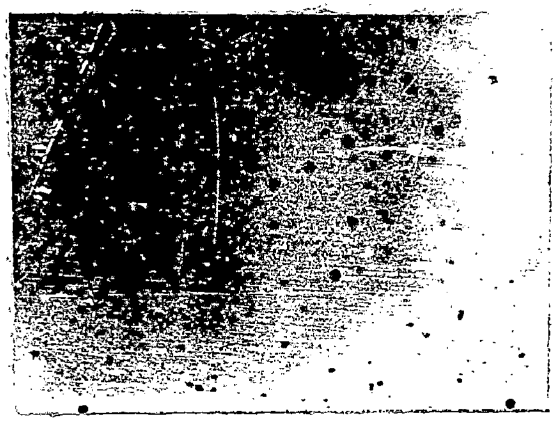

(a) (b) (c) (d)

Fig. 5·7 : Temperature 1200 °C
(a) 50 x (b) 200 x , 1hr · W·Q
(c) 200 x (d) 400 x , 3hr · W·Q

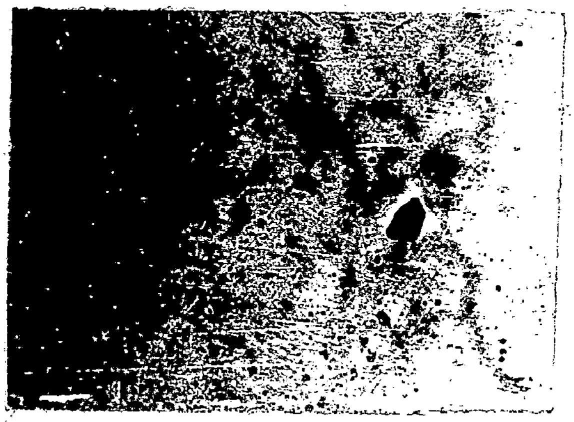
Fig. 5.8 Microstructure of inclusions



100X



200X



200X

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

AISI Grade[a]	Composition[b], %							
	Cr	Ni	C	Mn	Si	P	S	Other
301	16-18	6 - 8	0.15	2.0	1.0	0.045	0.030	--
302	17-19	8 -10	0.15	2.0	1.0	0.045	0.030	--
302B	17-19	8 -10	0.15	2.0	2-3	0.045	0.030	--
303	17-19	8 -10	0.15	2.0	1.0	0.20	0.15^c	Mo 0.60^d
303Se	17-19	8 -10	0.15	2.0	1.0	0.20	0.060	Se 0.15^c
304	18-20	8 -10.5	0.08	2.0	1.0	0.045	0.030	--
304L	18-20	8 -12	0.03	2.0	1.0	0.045	0.030	--
304N	18-20	8 -10.5	0.08	2.0	1.0	0.045	0.030	N 0.10-0.16
305	17-19	10.5-13	0.12	2.0	1.0	0.045	0.030	--
308	19-21	10 -12	0.08	2.0	1.0	0.045	0.030	--
309	22-24	12 -15	0.20	2.0	1.0	0.045	0.030	--
309S	22-24	12 -15	0.08	2.0	1.0	0.045	0.030	--
310	24-26	19 -22	0.25	2.0	1.5	0.045	0.030	--
310S	24-26	19 -22	0.08	2.0	1.5	0.045	0.030	--
314	23-26	19 -22	0.25	2.0	1.5-3.0	0.045	0.030	--
316	16-18	10 -14	0.08	2.0	1.0	0.045	0.030	Mo 2.0 -3.0
316F	16-18	10 -14	0.08	2.0	1.0	0.20	0.10^c	Mo 1.75-2.50
316L	16-18	10 -14	0.03	2.0	1.0	0.045	0.030	Mo 2.0 -3.0
316N	16-18	10 -14	0.08	2.0	1.0	0.045	0.030	Mo 2-3,N 0.1-0.16
317	18-20	11 -15	0.08	2.0	1.0	0.045	0.030	Mo 3.0 -4.0
317L	18-20	11 -15	0.03	2.0	1.0	0.045	0.030	Mo 3.0 -4.0
321	17-19	9 -12	0.08	2.0	1.0	0.045	0.030	Ti 5xC^c
330	17-20	34 -37	0.08	2.0	0.75-1.5	0.040	0.030	--
347	17-19	9 -13	0.08	2.0	1.0	0.045	0.030	Cb+Ta 10xC^c
348	17-19	9 -13	0.08	2.0	1.0	0.045	0.030	Cb+Ta 10xC^c Ta 0.1^e; Co 0.20^e
384	15-17	17 -19	0.08	2.0	1.0	0.045	0.010	

AISI Grade	Composition[a], %								
	Cr	Ni	C	Mn	Si	P	S	N	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	0.030	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
216[b]	17.5-22	5-7	0.08	7.5-9	1.0	0.045	0.030	0.50	3Mo
216L[b]	17.5-22	5-7	0.03	7.5-9	1.0	0.045	0.030	0.50	3Mo

[a] Balance iron. Single values are maximum values unless otherwise noted.
[b] Not standard AISI grades.

Sources: Source Book on Stainless Steels, American Society for Metals, 1976.
Allegheny Ludlum Alloy Data.

Table2.2 : Mechanical properties of the 300 and 200 series of Austenitic Stainless Steels.

AISI Grade	Tensile Strength MPa[c]	Yield Strength (0.2% Offset), MPa	Elongation, %	Hardness, Rockwell B
301	758	276	60	85
302	620	276	50	85
302B	655	276	55	85
303[b]	620	241	50	76
303Se[b]	620	241	50	76
304	586	241	55	80
304L	517	193	55	79
304N	620	331	50	85
305	586	262	50	80
308	586	241	50	80
309	620	310	45	85
309S	620	310	45	85
310	655	310	45	85
310S	655	310	45	85
314	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
321	620	241	45	80
330[b]	586	310	40	80
347	655	276	45	85
348	655	276	45	85
384	517	241	55	70

AISI Grade	Tensile Strength MPa	Yield Strength (0.2% Offset MPa)	Elongation %	Hardness, Rockwell B
201	758	379	55	90
202	689	379	55	90
205	862	482	58	98
216[c]	689	379	45	92
216L[c]	689	379	45	92

[a] Annealed sheet and strip.
[b] 1 MPa = 145.03 psi.
[c] Not standard AISI grades.
Sources: Source Book on Stainless Steels, American Society for Metals, 1976.
Allegheny Ludlum Alloy Data.

T2

Table 2.8 : Mechanical properties of the 400 series of Martensitic Stainless Steels.

AISI Grade	Condition	Tensile Strength MPa	Yield Strength (0.2% Offset) MPa	Elongation,%	Hardness, Rockwell B or C
403, 410, 416, 416Se	A[b]	517	276	30	B 82
	H[c]+T at 205°C	1310	1000	15	C 41
	316°C	1241	965	15	C 39
	538°C	1000	793	20	C 31
414	A	827	655	17	C 22
	H[c]+T at 205°C	1379	1034	15	C 43
	316°C	1310	1000	15	C 41
	538°C	1000	827	20	C 34
420, 420F	A	655	345	25	B 92
	H[d]+T at 316°C	1506	1345	8	C 50
422	A	793	586	22	B 98
	H[c]+T at 427°C	1627	1282	10	--
	538°C	1476	1145	13	C 42
	649°C	1000	862	14	C 32
431	A	862	655	20	C 24
	H[d]+T at 205°C	1413	1069	15	C 43
	316°C	1345	1034	15	C 41
	538°C	1034	896	18	C 34
440A	A	724	414	20	B 95
	H[d]+T at 316°C	1793	1655	5	C 51
440B	A	738	429	18	B 96
	H[d]+T at 316°C	1930	1862	3	C 55
440C, 440F[a]	A	758	448	13	B 97
	H[d]+T at 316°C	1965	1895	2	C 57

[a] Non-standard grade.
[b] A = annealed.
[c] H = hardened by heating to 982°C and cooling.
[d] H = 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.

Designation	Typical Composition[a], (%)							
	Cr	Ni	C	Mn	Si	P	S	Other
AISI Type 329	28.0	6.0	0.10	2.00	1.0	0.04	0.03	Mo 1.5
326[b]	26.0	6.5	0.05	1.00	0.6	0.01	0.01	Ti 0.25
SANDVIK 3RE60	18.5	4.5	0.02	1.50	1.6	0.01	0.01	Mo 2.5

[a] Balance iron.
[b] Earlier version known as developmental alloy IN-744; commercially available since 1970 under proprietary designations of UNILOY 326, AL 326 and H-326.
Sources: Various.

Table 2.10 : Mechanical properties of some Duplex Stainless Steels.

Designation	Tensile Strength MPa[b]	Yield 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	92

[a] Mill annealed condition.
[b] 1 MPa = 145.03 ksi.
Sources: Various.

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

AISI Grade	Composition^{a}, (%)						
	Cr	C	Mn	Si	P	S	Other
403	11.5-13	0.15	1.0	0.5	0.040	0.030	--
410	11.5-13.5	0.15	1.0	1.0	0.040	0.030	--
414	11.5-13.5	0.15	1.0	1.0	0.040	0.030	Ni 1.25-2.50
416	12 -14	0.15	1.25	1.0	0.060	0.15^{b}	Mo 0.60^{d}
416Se	12 -14	0.15	1.25	1.0	0.060	0.060	Se 0.15^{b}
420	12 -14	0.15^{b}	1.0	1.0	0.040	0.030	--
420F	12 -14	0.38	1.25	1.0	0.060	0.15^{d}	Mo 0.60^{d}
422	11 -13	0.20-0.25	1.0	0.75	0.025	0.025	Ni 0.50-1.0, Mo 0.75-1.25, W 0.75-1.25, V 0.15-0.3
431	15 -17	0.20	1.0	1.0	0.040	0.030	Ni 1.25-2.50
440A	16 -18	0.60-0.75	1.0	1.0	0.040	0.030	Mo 0.75
440B	16 -18	0.75-0.95	1.0	1.0	0.040	0.030	Mo 0.75
440C	16 -18	0.95-1.20	1.0	1.0	0.040	0.030	Mo 0.75
440F^{c}	17	1.0	0.4	0.4	0.04	--	S 0.08 or Se 0.18

[a] Balance iron. Single values are maximum values unless otherwise noted.
[b] Minimum.
[c] Non-standard grade, typical composition.
[d] Optional.
Source: Source Book on Stainless Steels, American Society for Metals, 1976.

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

AISI Grade	Composition[a], (%)						
	Cr	C	Mn	Si	P	S	Other
405	11.5 -14.5	0.08	1.0	1.0	0.040	0.030	Al 0.10-0.30
409	10.5 -11.7	0.08	1.0	1.0	0.045	0.045	Ti 6xC[d],0.75[c]
429	14 -16	0.12	1.0	1.0	0.040	0.030	--
430	16 -18	0.12	1.0	1.0	0.040	0.030	--
430F	16 -18	0.12	1.25	1.0	0.060	0.15[d]	Mo 0.6[e]
430FSe	16 -18	0.12	1.25	1.0	0.060	0.060	Se 0.15[d]
434	16 -18	0.12	1.0	1.0	0.040	0.030	Mo 0.75-1.25
436	16 -18	0.12	1.0	1.0	0.040	0.030	Cb+Ta 5xC[d],0.7[c],Mo 0.75-1.25
439[b]	17.75-18.75	0.07	0.60	0.60	0.040	0.030	Ti 12xC[d],1.0[c],Ni 0.5
442	18 -23	0.20	1.0	1.0	0.040	0.030	--
446	23 -27	0.20	1.5	1.0	0.040	0.030	N 0.25

[a] Balance iron. Single values are maximum values unless otherwise noted.
[b] 439 is not a standard AISI grade.
[c] Maximum.
[d] Minimum.
[e] 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 MPa[b]	Yield Strength MPa	Elongation,%	Hardness, Rockwell B
405	482	276	30	80
409	469	276	25	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	365	23	83
439[c]	482	280	30	80
442	551	345	20	90
446	551	345	25	86

[a] Annealed material.
[b] 1 MPa = 145.03 psi.
[c] 439 is not a standard AISI grade.
Source: Source Book on Stainless Steels. American Society for Metals, 1976.

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

Alloy	Typical Composition^{a}, (%)					
	Ni	Cr	Fe	C	Co	Ti
AISI Type 304^{b}	10	19	69	0.06	0.15	0.008
Alloy 800^{c}	33	21	38	0.03 max.	0.10 max.	0.60 max.^{f}
INCONEL Alloy 600^{d}	76	15	8	0.04	0.03	0.25
INCONEL Alloy 690^{e}	60	30	9.5	0.03	0.03	0.25

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

Alloys	Typical Composition (Wt. %)							
	Ni	Cr	Fe	Mo	C	Cb	Cu	Other
AISI Type 317	13.0	19.0	64.0	3.5	0.05	--	--	--
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 777	25.0	21.0	45.0	4.6	0.02	0.3	2.5	--
UDDEHOLM 904L	25.0	20.0	49.0	4.5	0.02	--	1.5	--
HAYNES No. 20 (Mod)	26.0	22.0	45.0	5.0	0.03	--	--	Ti 4xC
CARPENTER 20Cb-3	33.0	20.0	41.0	2.0	0.03	0.8	3.0	--
INCOLOY Alloy 825	42.0	20.0	30.0	3.0	0.03	--	2.0	--
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	--	--

Type of steel	Rupture stress (5 min), tsi	
	700°C	800°C
0.14 % C 14 % Cr 14 % Ni 2-3 % W	28.9	17.5
0.4 % C 8-12 % Cr 3 % Si	11.9	4.1

Table 2.12 Comparison of properties between an austenitic steel and a ferritic steel (11)

Symbol	C	Mn	Si	Ni	Cr	W	N
A	0.25	6	0.8	5	22		0.35
B	0.50	9	0.35	4	21		0.50
C	0.30	1.3	1.0	12	21		0.04
D	0.22	1.3	1.15	12	22		0.35
E	0.50	1.2	3	8	19	1.1	0.05

Table 2.14 Average mechanical properties of the austenitic valve steels in Table 2.13 (11)

Symbol	0.2% proof stress, tsi		Tensile strength, tsi		Elongation, %		R. of A. %		Mesnager impact, kg/cm^2	
	1*	2*	1	2	1	2	1	2	1	2
A	36	14	56½	24		18		25	1	10
B	48	20	70	27		5		10	1	3
C	23½	12	51	17	32	25		35	9	10
D	38	18	62	24	25	10		16	3	10
E	27	15	56	18		36		55	4	7

* 1 at room temperature; 2 at 750°C.

Table 2.13 Austenitic Valve Steels (11)

Table 5.1 Chemical composition of experimental material (27)

Element	C	Si	Mn	Cr	Ni	N
%	0.52	0.13	8.80	20.80	3.60	0.45

Table 5.2 Effect of Heat Treatment on the Hardness of Semi-rolled material in Air Cooled Condition

Temperature ^{0}C	Soaking Period	Hardness HV$_{30}$	Mean	S.D.
1150	1hr	379,383,360,336,358 346,398,363,379,398	369.90	19.8
	2hr	396,396,393,391,397 398,393,381,396,391	392.20	4.83
	3hr	334,371,370,375,373 387,377,404,383,387	376.10	17.06
1175	1hr	377,371,391,360,336 358,362,360,391,362	366.8	15.6
	2hr	425,413,383,391,387 402,439,404,409,436	408.9	18.5
	3hr	383,400,465,422,411 420,413,468,402,436	412.0	25.6
1200	1hr	336,339,331,346,329 326,334,344,343,328	335.6	6.6
	2hr	346,346,314,318,341 339,339,337,358,362	340.0	14.3
	3hr	346,318,341,318,333 339,326,334,336,339	333.0	9.0

Table 5.3 Effect of Heat Treatment on Hardness of Semi-rolled material in Water Quenched Condition

Temperature ^0C	Soaking Period	Hardness VHN_{30}	Mean	S.D.
1150	1hr	251,245,248,251,339 334,305,319,302,375	291.90	36.8
	2hr	331,331,302,364,312 346,337,346,378,325	325.2	15.1
	3hr	231,225,229,226,231 309,239,278,278,278	252.40	28.7
1175	1hr	341,344,344,302,305 306,319,238,312,331	320.2	17.3
	2hr	305,308,309,312,315 302,317,304,302,305	307.9	5.0
	3hr	268,265,266,268,260 293,275,286,287	273.6	10.5
1200	1hr	305,318,325,326,316 322,329,329,325,316	321.1	7.0
	2hr	329,331,317,287,276 285,277,278,290,317	298.70	21.0
	3hr	278,293,295,251,252 241,263,242,241,248	260.4	19.9

Table 5.4 Effect of Heat-Treatment on Hardness of As-rolled material in water quenched condition

Temperature ^{0}C	Soaking Period	Hardness VHN_{30}	Mean	S.D.
1150	1hr	331,323,320,326,345 348,331,347,331,348	335.20	10.6
	2hr	307,302,309,304,308 307,294,305,315,308	305.90	5.1
	3hr	305,307,311,308,301 304,313,314,338,315	311.60	8.1
1175	1hr	348,348,344,331,341 331,348,351,349,348	343.90	6.9
	2hr	329,326,331,321,325 329,331,329,328,308	327.70	2.8
	3hr	277,290,299,297,298 315,302,304,308,312	300.2	6.5
1200	1hr	311,304,274,307,298 304,304,305,297,304	302.80	4.7
	2hr	307,290,285,294,283 245,244,341,343,312	294.90	31.9
	3hr	315,315,343,331,321 305,303,304,308,314	306.80	35.3

Table 5.5

Mechanical Properties in the Solution Treated and Aged Condition

Soln. temp. °C	Soln. time	Ageing temp. °C	Ageing time	0.2% P.S. MPa	UTS Mpa	Elongation %	RA % (*)	Hardness Rc
1120	0.30	730	6.0	859.2	1193.05	6.05-12.03	11.2	33
1120	0.30	750	6.0	824.09	1135.01	9.87-10.9	11.3	32
1120	0.30	780	6.0	712.02	1090.78	3.82-10.8	11.4	32
1120	0.30	780	10.0	803.77	1166.17	10.39-16.14	10.8	34
1150	0.30	730	6.0	855.90	1171.01	4.9-11.28	10.5	37
1150	0.30	750	6.0	797.22	1187.98	5.54-13.58	11.5	34
1150	0.30	780	6.0	739.96	1162.97	6.54-15.49	12.0	33
1150	0.30	780	10.0	866.176	1280.98	15.58-20.56	11.5	35
1150	0.30	780	14.0	621.12	1132.38	15.27-19.09	12.0	36
1180	0.30	730	6.0	731.05	1125.87	6.92-12.39	11.5	31
1180	0.30	750	6.0	864.01	1208.36	4.76-11.15	10.9	32
1180	0.30	780	6.0	678.70	1043.14	5.49-12.39	11.0	34
1180	0.30	780	10.0	663.85	1048.04	11.81-12.17	10.8	31
1180	0.30	780	14.0	628.23	1160.26	19.02-20.98	10.5	30
1120	0.30	750	16.0	731.05	1121.54	14.71-17.11	10.4	33
1150	0.30	750	16.0	698.75	1065.4	12.18-14.77	10.8	32
1180	0.30	750	16.0	590.62	910.35	5.18-11.15	11.0	29

* R A as quoted; Experimentally found % elongation ~ 10%

** Typical composition; C=0.52, Si=0.13, Mn=8.80, Cr=20.80, Ni=3.60, N=0.45

Mechanical properties at room temperature as specified in the standard; Hardness=32 HRC, PS=580 MPa, UTS=950-1200 MPa, % EL=8, % RA=10

Table 5.6 XRD Analysis of As-Rolled Specimens

Heat treatment : 1150^0C 1 hr W.Q.

Sample	D.A. 2θ (°)	Fe_5C_2	Mn_5C_2	Fe_7C_3	Cr_7C_3	$Mn_{15}C_4$	Fe_3C	$(Cr,Fe)_7C_3$	$Cr_7C_3+Mn_7C_3$	Austeinte
1	54.8	(112)	(211)	(012)	(202)	(301)	-	-	-	-
	55.2	-	-	-	-	-	-	-	-	-
	55.4	(021)	(120)	-	-	-	-	-	-	(111)
	64.4	-	-	-	-	-	-	-	-	(200)
	64.6	(312)	(213)	(301)	(431)	-	-	-	-	-
	98.4	-	-	-	-	-	-	-	-	(222)

Table 5.7 XRD Analysis of As-Rolled Specimens
Heat Treatment : 1175^{0}C 3 hr W.Q.

Sample	D.A. 2θ (°)	Fe_5C_2	Mn_5C_2	Fe_7C_3	Cr_7C_3	$Mn_{15}C_4$	Fe_3C	$(Cr,Fe)_7C_3$	$Cr_7C_3+Mn_7C_3$	Austeinte
6	54.4	-	-	-	-	-	-	(202)	(555)	-
	55.0	-	-	-	-	-	(121)	-	(301)	-
	55.2	-	-	-	-	-	-	-	-	(111)
	64.4	-	-	-	-	-	-	-	-	(200)
	98.4	-	-	-	-	-	-	-	-	(220)

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Table 5.8 XRD Analysis of As-Rolled Specimens

Heat Treatment : 1200^0C 1 hr W.Q.

Sample	D.A. 2θ (°)	Fe_5C_2	Mn_5C_2	Fe_7C_3	Cr_7C_3	$Mn_{15}C_4$	Fe_3C	$(Cr,Fe)_7C_3$	$Cr_7C_3+Mn_7C_3$	Austeinte
7	54.4	-	-	-	-	-	-	(202)	(555)	-
	54.8	(112)	(211)	(012)	(202)	(301)	-	-	-	-
	55.4	(021)	(120)	-	-	-	-	-	-	(111)
	64.4	-	-	-	-	-	-	-	-	(200)
	64.6	(312)	(213)	(301)	(431)	-	-	-	-	-
	98.4	-	-	-	-	-	-	-	-	(222)

Table 5.9 XRD Analysis of As-Rolled Specimens

Heat Treatment : 1200^0C 3 hr W.Q.

Sample	D.A. 2θ (°)	Fe_5C_2	Mn_5C_2	Fe_7C_3	Cr_7C_3	$Mn_{15}C_4$	Fe_3C	$(Cr,Fe)_7C_3$	Cr_7C_3+Mn_7C_3	Austeinte
9	54.8	(112)	(211)	(012)	(202)	(301)	-	-	-	-
	55.0	-	-	-	-	(213)	(121)	-	-	-
	55.4	(021)	(120)	-	-	-	-	-	-	(111)
	64.4	-	-	-	-	-	-	-	-	(200)
	64.6	(312)	(213)	(301)	(431)	-	-	-	-	-
	98.4	-	-	-	-	-	-	-	-	(222)

Table 5.10 Nitrides in As-Rolled Heat Treated Specimens

Specimen No.	Heat Treatment	D.A.,(^{0}) 2θ	Fe-Nitride	Al-Nitride
(1)	1150^{0}C 1 hr, WQ	55.2 64.2 98.4	(101)	(102)
(6)	1175^{0}C 3 hr, WQ	55.2 64.2 98.4	(101)	(102)
(7)	1200^{0}C 1 hr, WQ	55.2 64.2 98.4	(101)	(102)
(9)	1200^{0}C 3 hr, WQ	55.2 64.2 98.4	(101)	(102)

Table 5.11 (a) Inclusion Rating Analysis

	Nature of Inclusions							
	A (sulfide)		B (Alumina)		C (Silicates)		D (Oxides)	
Specimen No.	Thin	Heavy	Thin	Heavy	Thin	Heavy	Thin	Heavy
1	✓	-	✓	-	-	-	✓	-
2	✓	-	✓	-	-	-	✓	-
3	✓	-	✓	✓	-	-	-	✓

Table 5.11 (b)
Typical Inclusion rating analysis*
Magnification : 100X

Filter Rating								
Specimen No.	**A (sulfide)**		**B (alumina)**		**C (silicate**		**D (oxides)**	
	Thin	**Heavy**	**Thin**	**Heavy**	**Thin**	**Heavy**	**Thin**	**Heavy**
1	1.0	-	1.5	-	-	-	1.5	-
2	1.5	-	1.0	-	-	-	1.5	0.5
3	1.0	-	1.5	-	0.5	-	1.5	-

* **Microscopes used :** Olympus

Area scanned ≈ 200 mm^2; Field Dia ≈ 0.8 mm

Data obtained from Ref. No. 23

Table 5.12

EPM Analysis of Inclusions Present

Element	Wt. (%)	to 100%	+/-	Atom	K-ratio
Si	0.014	0.359	0.013	0.6630	0.095
Al	0.902	3.458	0.021	6.6552	0.749
Fe	13.851	53.094	0.165	49.3666	15.005
Mn	5.119	19.621	0.136	18.5453	4.751
P	0.000	0.000	0.000	0.0000	0.000
S	0.775	2.971	0.157	4.8119	0.830
Cu	0.023	0.090	0.053	0.0786	0.022
Cr	4.191	16.060	0.098	16.0436	4.574
Ni	1.193	4.342	0.061	3.8408	1.085
Total	26.088	100.000		100.0000	27.110

ELEMENTAL SEGREGATION DATA BASED ON

Element	Wt. (%)	to 100%	+/-	Atom	K-ratio
Si	0.038	0.045	0.014	0.0884	0.039
Al	0.045	0.053	0.013	0.1080	0.036
Fe	52.851	62.300	0.319	61.2888	57.534
Mn	11.924	14.055	0.200	14.0562	11.158
P	0.000	0.000	0.000	0.0000	0.000
S	0.108	0.128	0.120	0.2186	0.116
Cu	0.000	0.000	0.000	0.0000	0.000
Cr	16.326	19.245	0.189	20.3348	18.206
Ni	3.540	4.173	0.095	3.9053	3.404
Total	84.832	100.000		100.0000	90.494

(1) TABLE- 5.13

Element	Wt. (%)	to 100%	+/-	Atom	K-ratio
Si	0.012	0.014	0.015	0.0275	0.012
Al	0.023	0.026	0.013	0.0529	0.018
Fe	52.959	61.055	0.320	60.0454	57.602
Mn	12.608	14.535	0.207	14.5319	11.801
P	0.010	0.011	0.044	0.0202	0.009
S	0.098	0.113	0.141	0.1930	0.105
Cu	0.000	0.000	0.000	0.0000	0.000
Cr	17.571	20.257	0.196	21.3970	19.524
Ni	3.460	3.989	0.096	3.7321	3.328
Total	86.740	100.000		100.0000	92.400

(7) TABLE-5.14

Element	Wt. (%)	to 100%	+/-	Atom	K-ratio
Si	0.060	0.070	0.015	0.1376	0.062
Al	0.000	0.000	0.000	0.0000	0.000
Fe	54.760	63.628	0.325	62.6965	59.614
Mn	10.757	12.499	0.192	12.5200	10.067
P	0.000	0.000	0.000	0.0000	0.000
S	0.000	0.000	0.000	0.0000	0.000
Cu	0.077	0.090	0.061	0.0778	0.072
Cr	16.653	19.350	0.192	20.4780	18.606
Ni	3.755	4.363	0.100	4.0901	3.611
Total	86.063	100.000		100.0000	92.031

(14) TABLE -5.15