

Thesis

On

**MECHANISMS FOR PRODUCING ULTRA-FINE/ NANO GRAINED
MICROSTRUCTURE FOR AUSTENITIC STAINLESS STEELS**

Submitted in partial fulfillment of the requirements for the award of degree

of

Master of Engineering
IN
PRODUCTION AND INDUSTRIAL ENGINEERING

Submitted By

Pankaj Sharma
Regd. No. 800982017

Under the Guidance of

Dr. Tarun Nanda
Assistant Professor
MED, TU, Patiala.

Dr. B. Ravi Kumar
Scientist E-II
MST Division, NML, Jamshedpur.

Dr. O.P. Pandey
Professor & Head
SPMS, TU, Patiala.



**DEPARTMENT OF MECHANICAL ENGINEERING
THAPAR UNIVERSITY**

(Established under section 3 of UGC Act, 1956)

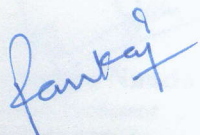
Patiala -147001, Punjab, India

July, 2011.

DECLARATION

I, Pankaj Sharma hereby declare that the work being presented in the thesis entitled, **'Mechanisms for producing Ultra-fine/ Nano Grained Microstructure for Austenitic Stainless Steels'** by me in partial fulfillment of the requirements for the award of degree of Masters of Engineering in Production and Industrial Engineering, from MED, Thapar University, Patiala is an authentic record of my own work carried under the supervision of Dr. Tarun Nanda, Assistant Professor, MED, TU, Patiala; Dr. O.P. Pandey, Head & Professor, SPMS, TU, Patiala and Dr. B. Ravi Kumar, Scientist E-II, MST Division, National Metallurgical Laboratory (NML), Jamshedpur.

The matter presented in this report has not been submitted in any other University/Institute for the award of Masters of Engineering or any other degree.



(Pankaj Sharma)

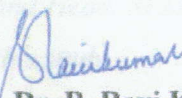
Date 12/7/11

CERTIFICATE

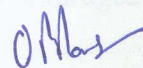
This is to certify that the thesis entitled, 'Mechanisms for producing Ultra-fine/ Nano Grained Microstructure for Austenitic Stainless Steels', being submitted by Pankaj Sharma (Regd. No: 800982017), in partial fulfillment of the requirements for the award of degree of Master of Engineering in Production & Industrial Engineering of Mechanical Engineering Department, Thapar University, Patiala, is a record of candidate's own work carried out by him under our supervision. To the best of our knowledge, the content of this thesis does not form a basis for the award of any previous degree to anyone else.



Dr. Tarun Nanda
Assistant Professor
MED, TU, Patiala.

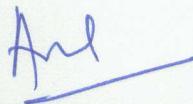


Dr. B. Ravi Kumar
Scientist E-II
MST Division, NML, Jamshedpur.



Dr. O.P. Pandey
Professor & Head
SPMS, TU, Patiala.

(Countersigned by)



Dr. Ajay Batish
Professor & Head
MED, TU, Patiala



Dr. S.K. Mohapatra
Dean of Academics Affairs
TU, Patiala

ACKNOWLEDGEMENT

I would like to express my deep sense of gratitude to Dr. B. Ravi Kumar, Scientist, Material Science & Testing division, National Metallurgical Laboratory (NML), Jamshedpur for his invaluable suggestions, excellent supervision, constant encouragement and critical discussion throughout the research work.

I take my opportunity to express my heartfelt adulation and gratitude to Dr. Tarun Nanda, Assistant Professor, Department of Mechanical Engineering, Thapar University, Patiala for his unreserved guidance, constructive suggestions, thought provoking discussions and unabashed inspiration in the nurturing work and during the preparation of manuscript.

My sincere thanks to Dr. Ajay Batish, Professor and Head, MED, TU, Patiala and Dr. S.K. Mohapatra, Dean of Academic Affairs, TU, Patiala for providing me with the opportunity to conduct this work and bring it out in the present form. I am grateful to Dr. O.P. Pandey, Professor and Head, SPMS, TU, Patiala who inspired, encouraged and supported me at all levels of this study.

I am grateful to Dr. Gautam Das, Mr. Manoj Kumar at NML, Jamshedpur, for allowing me to carry out the project work and permitting the use of necessary facilities for successful completion of the work.

I offer special regards to Ms. Sailaja, for providing her immense support in performing the experimental work throughout my stay at NML, Jamshedpur. I would also like to thank my friends specially Mr. Mridul Rai who have assisted me at all moments of need.

The greatest thanks go to my family for their infinite support. Above all, I express my indebtedness to the “ALMIGHTY” for all His blessing and kindness.

Pankaj Sharma

ABSTRACT

In the recent times the need for excellent mechanical properties of engineering materials for specific applications is realized. Austenitic stainless steels are very important as engineering materials. These are a large group of special alloys that have been developed, primarily, to withstand corrosion. Other desirable features may include excellent formability, room-temperature and cryogenic toughness, and good resistance to oxidation and creep at elevated temperatures. But these alloys show relatively low yield strength which is an area of considerable interest. Mechanical properties of steels strongly depend on their microstructure which is controlled by phase transformation, precipitation, recrystallization, and so on. Various methods have been adopted to enhance mechanical properties of these steels. The present work focuses mainly on improving the yield strength of austenitic stainless steels through grain refinement. The grain refining method includes thermo-mechanical treatment, which combines deformation and heat treatment. The method has been very effective for microstructural control and hence for improving the mechanical properties. In addition to thermomechanical treatment, thermal cycling process has also been developed in order to form ultrafine or nano-grained austenitic stainless steel. This helps to achieve a good combination of strength and ductility. Thermal cycling process consists of cold rolling and repetitive annealing to reduce the grain size. The study revealed suitability of thermal cyclic process for the production of ultrafine grained austenitic stainless steel having combination of high strength and good ductility.

LIST OF FIGURES

Figure No.	Description	Page No.
Figure 2.1	Various Grades of Stainless Steels	9
Figure 2.2	Pseudo-Binary Phase diagram	11
Figure 2.3	Schaeffler diagram	11
Figure 2.4	Fe-Ni phase diagram	14
Figure 2.5	Elongation to Strength Ratio for Various Steels	15
Figure 3.1	Rolling Mill for Thickness Reduction	33
Figure 3.2	Thermal Cycling Process	34
Figure 3.3	Tensile Specimen used in Study	35
Figure 3.4	Precision Cutter	37
Figure 3.5	Muffle Furnace	38
Figure 3.6	Ultrasonic Cleaner	39
Figure 3.7	Vickers Principle	39
Figure 3.8	Vickers Micro-hardness Testing Machine	40
Figure 3.9	Tensile Testing Machine	40
Figure 3.10	Cutting Machine	41
Figure 3.11	Mounting Press with Specification	42
Figure 3.12	Sample Mounting	42
Figure 3.13	Rotary Grinding Machine	43
Figure 3.14	Polishing Machine	44
Figure 3.15	Levelling Machine	45
Figure 3.16	Optical Microscope	46
Figure 3.17	XRD Machine	47
Figure 4.1	Stress - Strain Graph	50
Figure 4.2	Variation of Dislocation Density	51
Figure 4.3	Recrystallization Fraction with Annealing Time	52
Figure 4.4	Hardness Value with Cold Reduction	54
Figure 4.5	Dislocation Density vs Cold Reduction	55
Figure 4.6	Optical Image of 316L ASS at Different Temperatures	56
Figure 4.7	Recrystallization Fraction Vs Annealing Time	57
Figure 4.8	Microstructure of 316L Austenitic Stainless Steel	59
Figure 4.9	Microstructure of the Solution Annealed 316L SS	61
Figure 4.10	TEM Image of Cold Deformed Structure	61
Figure 4.11	TEM Images of Ultrafine Grained Microstructure	62
Figure 4.12	Stress Strain Curves for Solution Annealed and Cold Rolled Specimen	63
Figure 4.13	Stress- Strain curve for Thermal Cycled Specimen	64

CONTENTS

Title	Page No.
DECLARATION	i
CERTIFICATE	ii
ACKNOWLEDGEMENT	iii
ABSTRACT	iv
LIST OF FIGURES	v
CHAPTER 1 INTRODUCTION	1-7
1.1 General	1
1.2 Austenitic Stainless Steel	2
1.3 Grain Refinement in Austenitic Stainless Steel	3
1.3.1 Severe Plastic Deformation	4
1.3.2 Thermo-Mechanical Treatment	4
1.3.3 Thermal Cycling Process	5
1.4 Scope of Present Work	6
1.5 Organization of Thesis	6
CHAPTER 2 LITERATURE REVIEW	8-30
2.1 General	8
2.2 Crystallographic Texture	8
2.3 Stainless Steels	9
2.4 Grades of Stainless Steels	10
2.4.1 Ferritic Stainless Steels	12
2.4.2 Martenistic Stainless Steels	12
2.4.3 Austenitic Stainless Steels	12
2.4.4 Duplex Stainless Steels	13
2.4.5 Precipitation-Hardening Stainless Steels	13
2.5 Characteristics Features of Austenitic Stainless Steel	14
2.6 Strength Improvement in Austenitic Stainless Steels	16
2.7 Ultrafine Grained Material	17
2.8 Processing of Ultrafine or Nano Grained Material	17

2.8.1 Cold Rolling	18
2.8.2 Recrystallization	18
2.9 Effect of Stored Energy on Recrystallization Temperature	18
2.10 Review of Literature	19
2.11 Gaps in the Existing Approaches	29

CHAPTER 3 DESIGN OF THE STUDY	31-48
--------------------------------------	--------------

3.1 Introduction	31
3.2 Methodology	31
3.3 Establishment of Objective Function	32
3.4 Selection of Material	32
3.5 Experimental Procedure	33
3.6 Selection of Input Parameters	36
3.6.1 Amount of Cold Reduction	36
3.6.2 Annealing Temperature	36
3.6.3 Annealing Time	36
3.6.4 Number of Cycles	37
3.7 Machine and Testing Equipment	37
3.7.1 Precision Cutter	37
3.7.2 Muffle Furnace	37
3.7.3 Ultrasonic Cleaner	38
3.7.4 Vickers Micro Hardness Testing Machine	39
3.7.5 Tensile Testing Machine	40
3.8 Metallographic Specimen Preparation	41
3.8.1 Sectioning and Cutting	41
3.8.2 Mounting	41
3.8.3 Grinding	42
3.8.4 Polishing	43
3.8.5 Etching	44
3.8.6 Leveling	45
3.9 Microstructural Characterization	45
3.9.1 Optical Microscopy	45
3.9.2 X-ray Diffraction Technique	46

CHAPTER 1

INTRODUCTION

1.1 General

The strengthening of engineering metals and alloys through grain size reduction has long been a strategy through microstructure design. Studies of ultrafine grained / nano-grained materials in the past two decades have shown that grain refinement leads to substantial strengthening where abundant grain boundaries create obstacles to dislocation motion. With reference to the characteristics of polycrystalline materials, Ultra Fine Grained (UFG) materials may be defined as polycrystalline materials having very small grains with average grain sizes less than $\sim 1 \mu\text{m}$. Thus, the grain sizes of UFG materials lie within the sub-micrometer (100–1000 nm) and nano-meter (less than 100 nm) ranges. The processing, structure and properties of metallic materials with grain size in the range of ten to several hundreds of nanometre range have been of considerable interest (Valiev *et al.*, 2006).

Owing to the ultrafine microstructure, nanocrystalline materials are expected to exhibit superior mechanical properties compared with their coarse-grained counterparts, such as higher strength, more ductility and less strain-hardening effect. The deformation behaviour and microstructure development in nanocrystalline materials can be very different from those in conventional materials. These features, on one hand, are extremely interesting for extending our fundamental understanding of the structure–property relationship of materials when the structural modulation scale is reduced into the nano-meter regime. On the other hand, the unique properties of nanocrystalline materials may offer a wide range of technological applications in modern industries (Kumar *et al.*, 2003). From the application point of view, with the increasing number of application of nanocrystalline materials in microelectromechanical systems (MEMS), micro/nano devices, precision cutting tools, surface coating, and high performance structural applications. It is important to develop a detailed understanding of the intrinsic mechanical behaviour and underlying deformation mechanisms that govern the mechanical response of nanocrystalline materials. This fundamental knowledge is needed to be able to model and predict mechanical performance and to design for the use of nanocrystalline materials in various devices.

One of the most notable properties of nanocrystalline materials is their extremely high hardness and strength, which makes them attractive for structural applications where strength and weight are critical. Austenite Stainless steels have been exploited in a wide range of applications owing to their several desirable characteristics. They have excellent mechanical properties like combination of high strength and good ductility, good formability, and resistance to corrosion. Moreover, the austenitic microstructure renders the steel non-ferromagnetic. These properties of austenitic stainless steels make them highly desirable in many engineering applications (Lichtenfeld *et al.*, 2006). However, austenitic stainless steels are less suitable for structural applications due to their relatively low yield strength (230 to 350 MPa). Recent research and development studies have enabled wider use of austenitic stainless steels in structural applications with the increasing understanding of nanostructure and ultrafine grain polycrystalline materials. These materials have shown five to ten times increase in strength than their coarse grain counter parts (Eskandari *et al.*, 2009). The widespread enthusiasm for research on the mechanical behaviour or properties of nanocrystalline materials is driven by both scientific curiosity and this technological promise.

1.2 Austenite Stainless Steels

Austenitic stainless steels are the most popular type of stainless steels because of their excellent formability, corrosion resistance in various aggressive environments and good weldability. The presence of chromium (16-28%wt.), nickel (3.5-32%wt.) and small amount of the carbon (usually below 0.1%wt.) assure a stable austenitic structure in the whole range of the temperature (from the temperature of solidus to the room temperature). Moreover, it can contain such elements as molybdenum (2-6%wt.), titanium and niobium. They are widely used in chemical, petrochemical, machinery, automobile, nuclear and shipyard industries (Chowdhury *et al.*, 2005). Austenitic stainless steels are used in the production of a wide variety of formed and drawn parts for architectural, automotive, industrial, and domestic applications. Austenitic stainless steels can be classified as stable and metastable with respect to the stability of the austenite in them. One of the possible mechanisms of the plastic deformation in metastable austenitic steels is strain-induced martensitic transformation leading to the transition of paramagnetic austenite into ferromagnetic martensite. Austenite has a FCC crystal structure, while martensite at low carbon concentration crystallizes in BCC

structure. Martensite is harder and stronger than austenite. Some alloying components are inhibitors of martensitic transformation in austenitic stainless steels like N, Mn, and Cu.

Austenitic stainless steels are commonly chosen for applications where good corrosion resistance properties, moderate strength, and good aesthetic appearance are important. They are amenable to producing thinner and more durable structures and can also be formed into any desirable shape. The development of newer cost effective and high quality products are gaining importance due to the opening of opportunities in power generation sector, such as nuclear, solar, bio fuel power generation etc (Salishchev *et al.*, 2006)

1.3 Grain Refinement in Austenitic Stainless Steels

It is well known that mechanical properties of austenitic stainless steels are very sensitive to the chemical composition (which can induce hardening by both substitutional and interstitial solid solution) and to grain size. Hence, nitrogen alloying and cold rolling are methods generally applied to increase the strength. Recent developments have been carried out in stainless steels taking advantage of changes in the chemical composition induced by nitrogen addition. Nitrogen alloying is quite beneficial in increasing the strength by solid-solution strengthening. However, there is a low limit for nitrogen solubility in conventional grades and problems with hot ductility may also appear with increased nitrogen content. Cold rolling is a technique applied industrially to increase the strength. The yield strength of austenitic stainless steels, and particularly that of metastable alloys, can be drastically improved by cold deformation that generates strain-induced martensite in addition to ordinary strain hardening of high alloyed austenite. However, ductility is consequently decreased (Somani *et al.*, 2009). There are several mechanisms that have been successfully used to improve mainly the strength of austenitic stainless steels. These include solid solution strengthening, dislocation strengthening, precipitation strengthening, phase transformation strengthening and strengthening by grain refinement. Grain refinement is the most effective way to increase yield strength without impairing ductility. Since austenitic stainless steels do not undergo phase transformation at the typical annealing temperatures, the only way to refine the grains is either by dynamic recrystallization imposing very severe deformations (uniaxial or multiaxial) or by thermo-mechanical processes including both deformation and phase transformation to the degree of nano-structure or ultrafine grains (Di Schino *et al.*, 2002).

In the recent past, new approaches have been adopted to enhance the properties by reducing the grain size to nano-structures/grain and ultrafine-grain in polycrystalline materials. These involve reduction in grain size through various mechanisms like severe plastic deformation (SPD), thermo-mechanical treatment and thermal cycling processes etc (Karjalainen *et al.*, 2008). A fine grained material is harder and stronger than its coarse grained counterpart, since the former has a greater total grain boundary area to impede dislocation motion.

1.3.1 Severe Plastic Deformation

Severe Plastic Deformation (SPD) of metastable alloys is commonly accompanied by development of diffusion-free phase transformations that substantially modify the properties of materials. Severe plastic deformation techniques are commonly used laboratory methods to produce bulk nanocrystalline materials. Interest in the processing of bulk ultrafine-grained materials through the application of severe plastic deformation has grown significantly over the last decade. These techniques include high-pressure torsion, equal channel angular pressing, accumulative roll bonding, and bidirectional compression (Kawasaki, M. 2010). Processing by SPD refers to various experimental procedures of metal forming that may be used to impose very high strains on materials leading to exceptional grain refinement (Valiev *et al.*, 2006).

1.3.2 Thermo-Mechanical Treatment

Thermo-Mechanical Treatments (TMT) are large-scale industrial processes and can be more readily optimized as compared to SPD techniques. A recent novel route of forming ultra fine grained/ nano grained material involves annealing followed by large strain deformation. Large scale deformations promote rapid formation of many intersecting dislocation boundaries in different directions because of the variation in the slip systems operating from pass to pass. A gradual rise in misorientation across the strain-induced sub-boundaries with increasing strain finally leads to the evolution of ultra fine grains with high-angle boundaries. Materials processed by large strain deformation are characterized by high internal stresses, which are associated with both high dislocation densities and non-equilibrium grain boundaries. It is particularly important to study the annealing mechanisms operating in the ultra fine-grained metallic materials during heating (Tadashi, M. 1997).

In the context of obtaining high strength–high ductility combinations, a novel processing route of developing nano-grained/ultrafine-grained structure in metastable austenitic stainless steels involves controlled phase reversion annealing of cold deformed austenite. In this approach, severe deformation (~45 to 75 percent) of metastable austenite at room temperature leads to strain induced transformation of austenite to martensite. On annealing, this severely deformed strain-induced martensite reverts back to austenite either via a martenistic shear or diffusional reversion mechanism. Based on the percentage of cold deformation and annealing temperature-time sequence, different combinations of high strength–high ductility can be achieved. The optimal phase reversion annealing sequence results in the structure that is characterized by a combination of ultrahigh yield strength and excellent elongation of 800 - 1000 MPa and 30 - 40 percent, respectively. In recent years, there has been a renewed interest in the reversion transformation technique following severe cold deformation for developing NG/UFG structures in several austenitic stainless steels including 304, 304L, 301, and 316L (Misra *et al.*, 2010).

1.3.3 Thermal Cycling Process

Thermal cycling process is a temperature modulation process which consists of repetitive cold rolling and annealing to reduce grain size. The basic mechanism of grain refinement by cyclic thermal process may be hypothesized as (Kumar *et al.*, 2009):

- Nucleation preferentially occurs in high strain energy regions during the first few thermal cycles and sets in increased strain heterogeneity between the recrystallized and residual deformed regions.
- On subsequent thermal cycles, nucleation takes place in the residual deformed region. These regions are the more potential sites owing to their high stored strain energy, in preference to the recrystallized region.
- The driving force for the high energy grain boundary migration for grain growth of the recrystallized grains is small due to inherent sluggish diffusion rates and mobility of the high angle grain boundaries in austenite.

1.4 Scope of Present Work

Several major drivers have contributed to the rapid increase in research into nanocrystalline metals and alloys in recent years. Most of the academic writings have focused on formation of ultrafine austenitic grains in considerably metastable austenitic stainless steels in the presence of strain induced martensite (SIM). However, the literature lacks in availability of reports on grain refinement of austenite to submicron size in the absence of SIM. The mechanical behaviour and evolution of phases in metastable materials, particularly Fe-Cr-Ni steels, have been the subject of experimental and theoretical studies for a long time. The reason is that these materials are quite often used in modern engineering. However, their mechanical properties are not fully investigated, because the character and the intensity of phase transformations depend on many factors, such as strain rate and strain level, stress state and regime of mechanical loading, and temperature. Thus, the main focus of present research is to investigate formation of ultrafine grains in less metastable austenitic stainless steels. Also a limiting factor for the implementation of thermo-mechanical processes in less metastable austenitic stainless steel is the absence of phase transformation in these steels. The present research work intends to investigate the important microstructural changes occurring in cold deformed austenite grains during thermal cycling in the absence of phase transformations (austenite to SIM). Further, it attempts to predict the factors affecting kinetics of recrystallization during thermal cycling process.

1.5 Organization of the Thesis

The write up of the thesis is divided into five chapters as discussed as follows:

- **Chapter I** justifies the need of present work. It underlines the aim of research work with future scope of work and covers the organization of thesis.
- **Chapter II** reviews in detail the previous studies in the field to identify the factors which facilitate. It identifies the basic mechanisms to understand the research problem. The chapter identifies the main limitations in the existing academic writings. The chapter also discusses the research hypothesis.
- **Chapter III** introduces overall design of the study. It explains the methodology adopted for carrying out the research work. The details of work done in each phase of research, the tools and techniques used have been presented.

- **Chapter IV** presents the overall results and discussions for the present study. The chapter also identifies empirical relation used for calculations and validation of experimental results.
- **Chapter V** covers the summary of research work, its results, conclusions, and the recommendations. The scope for future work has also been covered in subsequent sections of this chapter.

CHAPTER 2

LITERATURE REVIEW

2.1 General

This chapter provides literature based background information for the present investigation. The first section identifies the necessity of assessment of crystallographic texture, and reviews the properties and grade of material under consideration. It also discusses the characteristic features of austenitic stainless steels. The next section presents a detailed review of literature and results of experimental work conducted in the subject area.

2.2 Crystallographic Texture

Texture, the non-statistically distributed orientation of single crystal of a polycrystalline material, has been known to be present in minerals, metals and alloys for a long time. The origin of non-random orientation distribution lies in the crystallographic structure of the crystal and the orientation relationship between the crystal and its neighbouring crystals. It influences several important properties of engineering materials viz. mechanical, electrical, and magnetic and also the behaviour of materials like formability, corrosion and others. In fact, there exist two groups of material properties: the ones which do not depend on crystallographic orientation of the material and others which depend on it. For example, both mass and density are independent of the crystallographic orientation of a material whereas electrical conductivity and magnetization depend on the crystallographic orientation (if the material is electronically or magnetically conductive). When a material develops texture, a crystallographic preferred orientation is formed with respect to the geometrical orientation of the sample and the sample has anisotropy in properties that are a function of its crystal orientation. Texture is developed at all stages of the manufacturing process of metals/alloys but the precise nature of texture is a complex function of mechanical and thermal treatment as well as the material itself. This explains why texture analysis is critical to the understanding of properties of polycrystalline materials and the relationship between properties and process parameters (that lead to the formation of texture). By investigating texture formation at different process conditions, the origin of anisotropy in properties may be unravelled and this

information is precious for further development of materials. The results from texture analysis can provide clues to how properties and processes are related through microstructure evolution.

2.3 Stainless Steels

Stainless Steels (SS) form some of the most important alloy systems among various ferrous alloy groups. This is owing to their excellent corrosion resistance, good formability, a broad range of strength levels and pleasing appearance, which are not attainable with other ferrous alloy groups. Stainless steels are iron-based alloys having a minimum of 11% Cr which is essential to bring about the required levels of corrosion and oxidation resistance to the steel. These alloys form invisible and adherent chromium rich oxide films on the surface which renders it the characteristic stainlessness. In addition to Cr, other alloying elements such as Ni, Mo, Cu, Ti, Nb, etc. are often made to make profound changes in the structure and properties. Carbon is usually present in amounts less than 0.03% to over 1.0% depending on type of stainless steel. A summary of various grades of stainless steels along with their alloying elements is presented in the Figure. 2.1 (Davis, 1994).

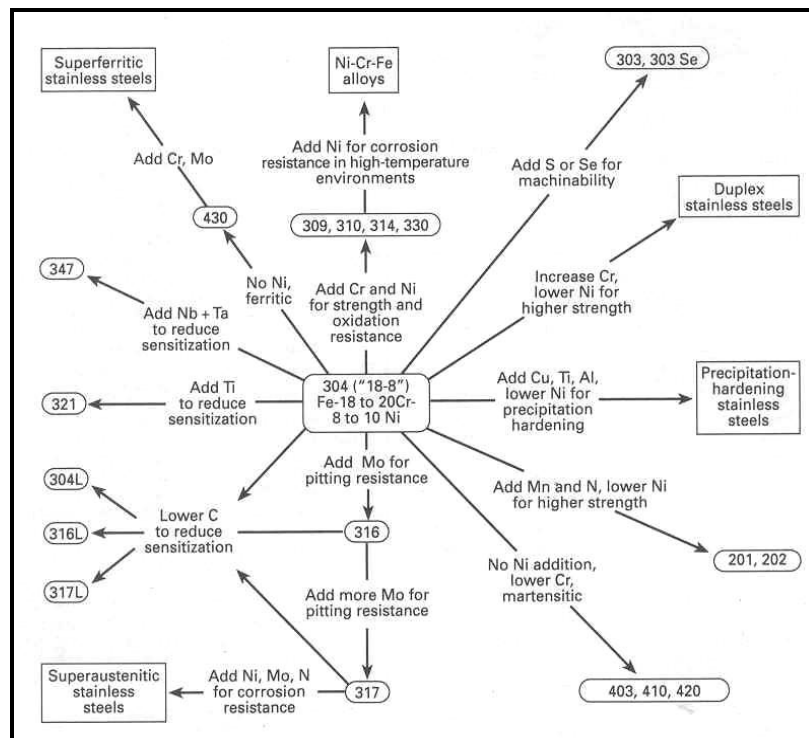


Fig. 2.1 Various Grades of Stainless Steels

Over the years, stainless steels have become an important class of engineering alloys, used for a wide range of applications and in many environments. They are extensively used as cooking utensils, fasteners, equipments for use in chemical plants, dairy and food processing plants, health and sanitation applications, petrochemical and petroleum plants, textile plants, pharmaceutical and transportation industries and power generation. Some of these applications involve exposure to elevated or cryogenic temperatures. Depending on applications, the selection of proper type of stainless steel has to be made. Corrosion resistance is frequently the most important characteristic of a stainless steel considered for a specific application.

2.4 Grades of Stainless Steels

Basically, stainless steels are based on the iron-chromium, iron-chromium-carbon, and iron-chromium-nickel systems. Additionally they contain a number of alloying elements in order to control/modify their microstructure and properties. Based on alloy composition stainless steels are grouped into five major categories. The first four categories are based on characteristic crystallographic structure or microstructure of alloys in the family. The fifth family, the Precipitation-Hardenable alloys, is based on the type of heat treatment of austenitic stainless steels (and not on the basis of microstructure). The various grades with a typical example of each family are presented:

- I) Ferritic Stainless Steels [e.g. AISI 430SS (C = .08% max; Cr = 16-18%; Mn = 1.0%)]
- II) Martensitic Stainless Steels [e.g. AISI 403SS (C = 0.15% max, Cr = 11.5-13%, Mn = 1.0%)]
- III) Austenitic Stainless Steels [e.g. AISI 304SS (C = 0.08% max, Cr = 18-20, Mn 2.0%, Ni = 8-12%)]
- IV) Duplex (austenitic plus ferritic) Stainless Steels [e.g. AISI 329SS (C = 0.2% max, Cr = 23-28%, Mn = 1.0%, Ni = 2.5-5%)]
- V) Precipitation-Hardenable Stainless Steels [e.g. 17-4P H (C = 0.04% max, Cr = 16%, Mn = 0.3%, Ni = 4.2%, Cu = 3.4%)]

Figure 2.2 shows the Iron-chromium binary phase diagram which forms the foundation of austenitic stainless steels (Krivobok, 1993). On the other hand, Figure 2.3 shows the Schaeffler diagram showing different phases in various stainless steels (Pickering, 1976).

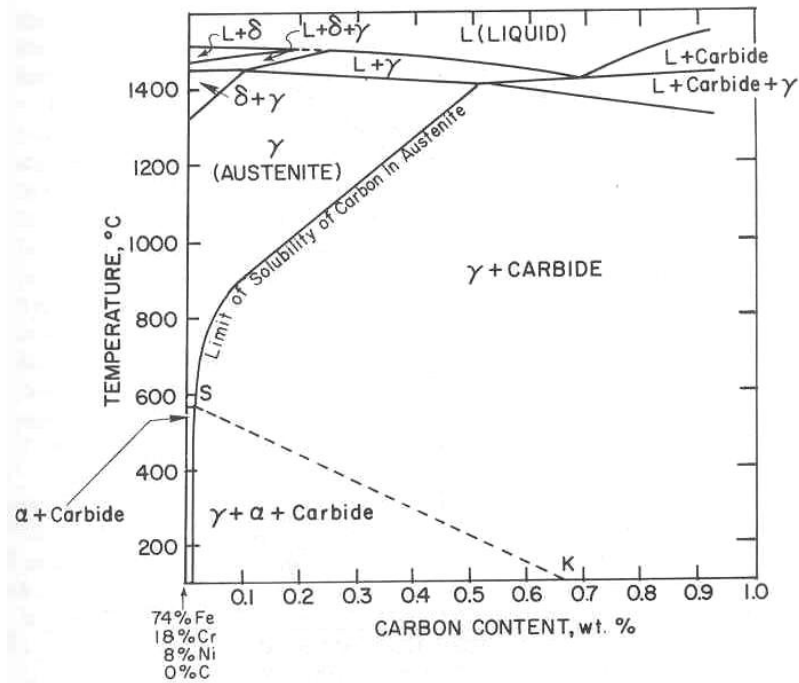


Fig. 2.2 Pseudo-Binary Phase Diagram for a typical Austenitic Stainless Steel

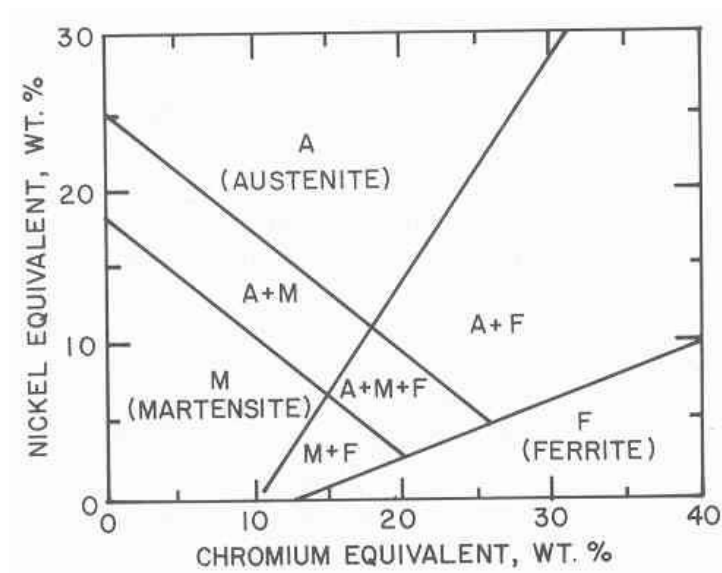


Fig. 2.3 Schaeffler Diagram Showing the Structure Related to the Composition of Stainless Steels

2.4.1 Ferritic Stainless Steels

Ferritic stainless steels are essentially chromium (10.5 to 30% Cr) containing alloys having BCC crystal structure. In general, these steels have high strength. They can have good ductility and formability, but high temperature strengths are relatively poor compared to austenitic grade. These steels show very good resistance to chloride stress-corrosion cracking, atmospheric corrosion and oxidation at relatively low cost. These steels have low strain hardening rates and any cold working significantly lowers their ductility. Ferritic stainless steels generally require rapid cooling from hot working to avoid grain growth and embrittlement. This restricts its use to relatively thin gages only. These alloys cannot be hardened by heat treatment as no phase transformation occurs (into harder phases like martensite etc). Ferritic stainless steels can offer moderate to outstanding corrosion resistance depending on alloy content. Due to their embrittlement and sigma-phase (approximately Fe-Cr) formation, long term service exposure of these steels is restricted to 250° C maximum.

2.4.2 Martenistic Stainless Steels

Martenistic stainless steels are alloys of chromium and carbon having Body Centered Tetragonal (BCT) structure. These can be hardened by heat treatment. The strength obtained by heat treatment depends on carbon content of the alloy. Martenistic stainless steels possess moderate corrosion resistance. These steels can be moderately hardened by cold working. In hardened and tempered condition these alloys have good ductility and toughness properties. These are used when the application demands good tensile strength, creep resistance, and fatigue strength properties, in combination of moderate corrosion resistance and heat resistance. Martenistic stainless steels are prone to temper embrittlement when heated in the range of 425-565° C.

2.4.3 Austenitic Stainless Steels

Austenitic stainless steels constitute the largest family in terms of number of alloys and usage. They attain their austenitic structure even at room temperature through the use of austenite stabilizing elements chiefly Ni. Other alloying elements viz. manganese, nitrogen and carbon are also used as austenite stabilizers. Austenitic stainless steels have face-centered cubic structure and are nonmagnetic in nature. Like ferritic alloys, austenitic alloys can also not be

hardened by heat treatment, which is evident from the phase diagram as shown in Figure 2.2. Upon cooling from high temperature austenite does not undergo any phase transformation which would result into increase in hardness. These steels possess excellent cryogenic properties and good high temperature strength. Though these can be hardened by cold working, but become magnetic by cold working due to formation of martensite. While heating in the temperature range of 425 - 870° C, austenitic stainless steels undergo sensitization, which occurs due to chromium carbide layer depletion at/near grain boundaries. Sensitization makes these steels susceptible to intergranular corrosion. Also the formation of sigma phase (chromium-molybdenum rich intermetallic phase) reduces the corrosion resistance of these steels. However, degraded corrosion resistance due to above two phenomena can be corrected by solution annealing treatment.

2.4.4 Duplex Stainless Steels

Duplex stainless steels are chromium-nickel-molybdenum alloys that are balanced to contain a mixture of austenite and ferrite phases. The volume fraction of these phases depends on the composition and heat treatment provided. Nitrogen, molybdenum, copper, silicon and tungsten are added to control structural balance. The duplex nature of structure enhances its stress-corrosion cracking resistance. The optimum phase balance in these alloys is usually equal amounts of ferrite and austenite. Duplex stainless steels are useful in intermediate temperature range where resistance to acids and aqueous chlorides is required. These alloys exhibit very high yield strength compared to ferritic and austenitic stainless steels which leads to major weight reduction in their application. Their high yield strength renders them difficult to cold forming compared to austenitic steels. Further, due to their susceptibility to high-temperature embrittlement, hot working requires more attention than austenitic steels.

2.4.5 Precipitation-Hardening Stainless Steels

Precipitation-hardening stainless steels are chromium-nickel alloys that can be hardened by an ageing treatment. In general, these steels attain high strength by precipitation hardening of the martensitic structure. Precipitation-Hardening stainless steels may be austenitic or martensitic in annealed condition and annealed austenitic phase is transformable to martensite through conditional heat treatments or sometimes subzero treatment. Depending on their martensite start and finish temperatures (M_s and M_f temperature) and behaviour on cooling,

these are grouped into martensitic, semi-austenitic and austenitic categories. Owing to their high strength (yield strength is in the range 515-1415 MPa), precipitation-hardening stainless steels find extensive use in aerospace and high-technology industries.

2.5 Characteristics Features of Austenitic Stainless Steels

Austenitic stainless steels are chromium steels with high nickel content together with silicon, manganese and other alloying elements designed especially for high corrosion resistance. These contain 17 to 25% chromium and 8 to 20% nickel. The amount of carbon is kept very low. Austenitic stainless steel forms the 2XX and 3XX series of American Iron and Steel Institute (AISI). The tendency of nickel to stabilize austenite is sufficient to overcome the ferrite stabilizing tendency of chromium. It should be noted that 18% Cr, 8% Ni falls just inside the γ region as shown in Figure 2.4 (Raghvan, 2004). At lower temperatures, γ should be transformed to α , but the transformation is very sluggish. As a result a steel containing 18% Cr, 8% Ni will be austenitic at all temperatures. However, cold working may cause austenitic stainless steel to transform a martensite reaction. Carbon and manganese tend to stabilise austenite while chromium (Cr), molybdenum (Mo), silica (Si), and niobium (Nb) stabilize ferrite. Nitrogen is a strong austenite stabilizer and has been used to substitute for a portion of the nickel.

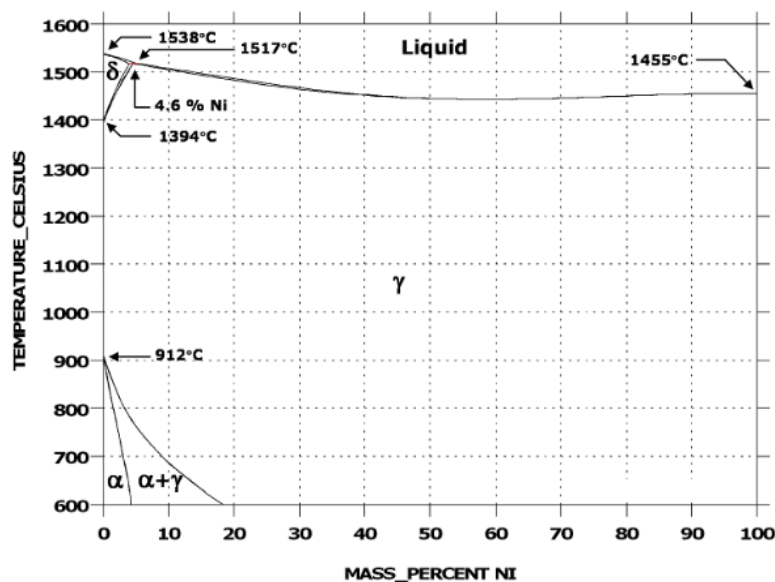


Fig 2.4 Fe-Ni Phase Diagram

Austenitic stainless steels have remarkable properties which clearly differentiate them from all the other categories of stainless steel. Austenitic stainless steels are commonly chosen for applications where good corrosion resistance properties, moderate strength, and good aesthetic appearance are important. They have good formability and are amenable to producing thinner and more durable structures and can also be formed into any desirable shape. However, austenitic stainless steels are less suitable for structural applications due to their relatively low yield strength (230 to 350 MPa). It is well known that mechanical properties of austenitic stainless steels are very sensitive to the chemical composition (which can induce hardening by both substitutional and interstitial solid solution) and to grain size. Hence, nitrogen alloying and cold rolling are methods generally applied to increase the strength. The yield strength of austenitic stainless steels, and particularly that of metastable alloys, can be drastically improved by cold deformation that generates strain-induced martensite in addition to ordinary strain hardening of high alloyed austenite. However, ductility is consequently decreased (Somani *et al.*, 2009). Another effective way to increase yield strength without impairing ductility is grain refining. Since austenitic stainless steels do not undergo phase transformation at typical annealing temperatures, the only way to refine the grains is either by dynamic recrystallization imposing very severe deformations (uniaxial or multiaxial) or by thermo-mechanical processes including both deformation and phase transformation to the degree of nano structure or ultrafine grains ((Di Schino *et al.*, 2002).

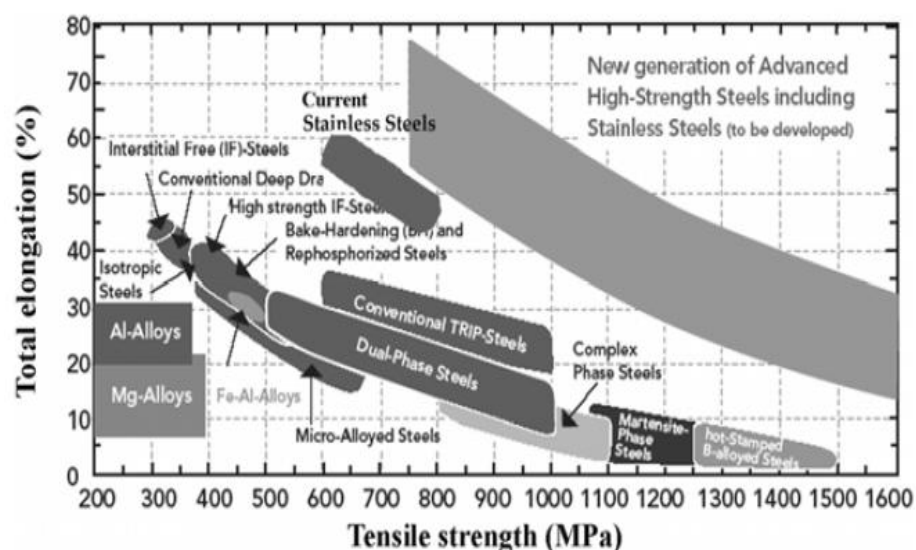


Fig 2.5 Elongation to Strength Ratio for Various Steels

Tensile strength of austenitic stainless steels is in the range of 600 - 800 MPa and total elongation from 45 to 60 % is investigated. In regard to the ratio of strength and elongation (as illustrated in Figure 2.5), however, stainless steels are located well above the ranges of carbon steels. Nevertheless, there is a great interest in improving the mechanical properties of commercially available austenitic stainless steels, so that these materials could be better utilized in structural applications and automotive industry (Karjalainen *et al.*, 2008).

2.6 Strength Improvement in Austenitic Stainless Steels

There are several mechanisms that have been successfully used to improve mainly the strength of austenitic stainless steels. These include Solid Solution Strengthening, Dislocation Strengthening, Precipitation Strengthening, Phase Transformation Strengthening and Strengthening by Grain Refinement. In case of solid solution strengthening, lattice distortion produced by solute atoms in the solvent atoms (of different sizes and diameters) offers resistance to dislocation movement and hence produces strengthening effect. Dislocation strengthening mechanism or strain hardening mechanism is an important industrial process that is used to harden metals or alloys that do not respond to heat treatment. This is a mechanism of increasing hardness and strength of a metal or alloy when subjected to plastic deformation at temperatures lower than the recrystallization temperature. The dislocation density increases with deformation, due to dislocation multiplication or formation of new dislocations. As the dislocation density increases, the resistance to dislocation motion by other dislocations becomes more pronounced. Precipitation strengthening is the strengthening of an alloy produced from precipitation of finely dispersed second phase from a super saturated solid solution. The essential condition for the precipitation to take place in solid solution is the decreasing solubility of a solute with decreasing temperature. Phase transformation strengthening is a mechanism to increase the hardness and strength by change in phase or microstructure of metal or alloy due to heating- quenching processes (Raghavan V., 2004).

Strengthening by grain refinement involves reduction in grain size through various mechanisms like severe plastic deformation, thermo-mechanical treatment and thermal cycling processes etc. A fine grained material (one that has small grain size) is harder and stronger than a relatively that is coarse grained, since the former has a greater total grain boundary area to impede dislocation motion.

Grain boundary region acts as a barrier to dislocation motion for two reason (Callister, 1997):

1. Since the two grains are of different orientation, a dislocation passing from one grain to other will have to change its direction of motion; this becomes more difficult as the crystallographic misorientation increases.
2. The atomic disorder within a grain boundary region will result in a discontinuity of slip planes from one grain into other.

2.7 Ultra Fine Grained Materials

Nanocrystalline and ultrafine grained materials exhibit superior performances and properties. These materials are attractive for many ranges of application from biomedical to aerospace industries. While high strength and good ductility rarely exist simultaneously in any material, UFG alloys exhibit the optimal combination between both. The general trend for coarse-grained (CG) materials demonstrates a trade-off between both properties: with decreasing the alloy grain size, its strength usually increases while ductility decreases. The former originates from the Hall Petch relationship as shown in Equation 2.1 (Callister, 1997):

$$\sigma_y = \sigma_o + \frac{K}{\sqrt{d}} \quad \text{Eq. 2.1}$$

Where σ_y is the measured yield strength, d is the grain size and σ_o and K are constants. An inverse Hall-Petch behaviour is observed below a critical grain size, which is in the nanoscale regime. On the other hand, ductility also deviates from its general trend in the UFG regime due to the operation of additional deformation mechanisms besides dislocation slip. The balance between both dependences implies that sub-micron grained alloys may possess high strength concurrently with good ductility.

2.8 Processing of Ultrafine or Nano Grained Material

Austenitic stainless steels, despite their excellent corrosion resistance, have low yield strength. One of the most effective ways to increase yield strength without impairing good ductility is by grain refinement. Austenitic stainless steels do not exhibit any transformation during hot deformations, so grain refinement can be achieved only by recrystallization. Hence ultrafine grains can be achieved in two steps which is cold rolling followed by recrystallization (Kumar *et al.*, 2003).

2.8.1 Cold Rolling

Deformation by cold rolling is a process by which the metal/alloy is introduced between rollers and then compressed and squeezed at room temperature (below the recrystallization temperature) for reducing its cross sectional area. It is convenient to express the degree of plastic deformation as percent cold work as follows:

$$\% \text{ cold work} = \frac{(A_0 - A_d)}{A_0} \times 100$$

Where A_0 = the original area of the cross section, and

A_d = the cross-sectional area after deformation

2.8.2 Recrystallization

Recrystallization (often called primary recrystallization) is the annealing process involving nucleation and growth of new, strain-free and equi-axed grains in the microstructure of a plastically deformed metal. The grain size of a recrystallized metallic material depends on the rates of nucleation and subsequent growth of the recrystallized grains. During recrystallization, the mechanical properties that were changed as a result of cold working are restored to their pre cold-worked values. The critical deformation (% cold-work) is the degree of deformation which is just necessary to initiate recrystallization. Recent research has shown that deformation rate should be raised to a given minimal rate for starting recrystallization depending on the kind of material. Thus, the energy required for the appearance of new grains can be provided. It has also been reported that if the energy and the grain number required for critical deformation are insufficient, a recrystallized matrix having coarse grains will be produced. In other words, the higher the cold deformation rate, the thinner the grains in the matrix. If the recrystallization temperature is increased, recrystallization can occur at lower cold deformation rates. The recrystallization temperature is reduced when the cold deformation rate is increased (Di Schino *et al.*, 2003).

2.9 Effect of Stored Energy on Recrystallization Temperature

In polycrystalline metals the stored energy of cold work plays a significant role in driving recrystallization. In general, the stored energy of cold work in metals arises from the evolution of a variety of defects including, twins, stacking faults, grain or sub-grain boundaries, as well as dislocations. Experimentally found changes in the stored energy are

caused on a microscopic level mainly by the generation, blocking and annihilation of dislocations, leading to changes in the density and arrangements of dislocations. The rate of accumulation of stored energy varies with the strain given to the material. At lower stored energy, a relatively fine grained material recrystallizes at a lower temperature than a coarse grained material of same stored energy. However, at higher stored energies, a coarse grain sized material recrystallizes at a lower temperature. Conversely at lower recrystallization temperatures, for a given recrystallization temperature, a lower stored energy is necessary in coarse grained material than in fine grained material. However the coarser grained material suffers much greater strain to reach the stored energy. At higher stored energies, the recrystallization temperature continues to decrease (as the strain increases) even though there is negligible or no increase in the stored energy of cold work (Baker *et al.*, 1994).

2.10 Review of Literature

This section presents a detailed review of existing academic writings for improving the strength and other properties of austenitic stainless steels by grain refining mechanisms:

Yamamoto *et al.* (1993) examined the strengthening mechanism and optimum strength control method, on an industrial scale, for stainless steels of type 304 austenitic stainless steels (nominal composition; Cr: 18-20%, Ni: 8-12%, Mn: 2%, C: max 0.08%) produced by thermo-mechanical control process (TMCP). It was found that grain-size dependence of yield strength in austenitic stainless steel was two thirds of that in plain carbon steel, so that strengthening by grain refinement through hot rolling at temperatures above 970° C was limited to 100 MPa. Therefore, for further strengthening, substructure strengthening through rolling in the region of no recrystallization at lower temperatures between 800° C and 900° C is necessary. The total reduction in the no recrystallization region is the key factor for controlling the strength and hence it is important to attain an aimed strength level (0.2% proof stress: 250-500 MPa) on an industrial scale.

Takaki *et al.* (1994) obtained ultra-fine austenite grains (with a submicron size) by a new thermo mechanical treatment, which applied reversion from deformation induced martensite in a metastable austenitic stainless steel. In this process, the solution treated metastable austenitic steel is heavily cold rolled (more than 80% reduction) to produce strain-induced martensite and further reversion of this martensite to austenite. To use the martensite to

austenite reversion effectively, majority of the initial austenite phase should transform to martensite after cold rolling. In the case of metastable austenitic stainless steels, induced martensite can revert to austenite by diffusional transformation at relatively low temperatures, around 900K, where the growth of reverted austenite grains is inhibited. Consequently, ultra-fine austenite grains can be obtained by this process.

Tadashi M (1997) studied various types of thermomechanical treatments, such as controlled rolling followed by accelerated cooling (i.e. a thermo-mechanical control process [TMCP]) and intercritical heat treatment in austenitic, ferritic, martensitic and duplex stainless steels in order to increase strength without sacrifice, of ductility, toughness and corrosion resistance. The process were very effective for microstructural control and hence for improvement of mechanical properties (to improve the strength-ductility (toughness) balance in addition to good corrosion resistance.)

Di Schino *et al.* (2002) showed the effect of martensite formation and austenite reversion on grain refining in AISI 304 stainless steels. It was observed that in a 30% deformed sample, some austenitic grains begin to nucleate in the martensitic microstructure; in a 70% deformed sample, martensite is almost completely consumed by the formation of new austenitic grains, but an inhomogeneous structure is evident; finally, when cold rolled reduction is increased upto 90%, the microstructure is fully recrystallized, the grain size distribution is more homogeneous and the average grain size is about $0.8 \mu\text{m}$. In the AISI 304 stainless steel, Hall-Petch relationships holds down to at least $0.8 \mu\text{m}$ of grain size. This result does not agree with that of similar measurements on AISI 301 stainless steel, where a deviation from the Hall-Petch relation is detected at about $3\mu\text{m}$ grain size. Further, for temperatures lower than 800°C , grain growth after recrystallization is not activated while, for higher temperatures, thermally activated grain growth occurs, with a subsequent temperature upper limit to grain refining.

Samuel *et al.* (2002) analyzed the influence of temperature and strain rate on tensile work hardening behavior of type 316 LN austenitic stainless steel. The analysis of tensile work hardening steel revealed a three-stage behavior. The variations of instantaneous work hardening rate $\theta = d\sigma/d\varepsilon$ and $\theta\sigma$ as a function of σ have been successfully used to interpret the tensile work hardening behaviour of type 316 LN austenitic stainless steel. The stage II

hardening is observed as a rising part insensitive to test temperature and strain rate. The anomalous variation in work hardening rate observed at intermediate temperatures is ascribed to dynamic strain ageing. Increased dynamic recovery at high temperatures resulted in a rapid decrease in work hardening rate.

Sahay *et al.* (2003) showed the accelerated grain growth behavior during cyclic annealing of cold rolled steel samples. In addition, several other observations, such as increase in grain size with cycle frequency and increase in grain size with increase in thermal amplitude were made in this study. It was concluded that several anomalies viz., accelerated grain growth, retardation of grain growth by introducing intermediate holds, increase in grain size with cycle frequency (keeping the T_{high} and T_{low} constant) and an increase in grain size with decrease in T_{low} which cannot be explained by quasi-isothermal grain growth kinetics, were observed during cyclic annealing. These anomalies were attributed to the influence of non-isothermality on grain boundary mobility and kinetics of grain growth, which is not captured by isothermal kinetics based theories.

Chia *et al.* (2005) described a dislocation density model which shows the relation between grain size and flow stress. The work discussed about Hall-Petch plot that is relatively independent of ε and T . The dislocation density (ρ) increases with ε and with decrease in d , giving the well-known relation $\sigma/M \approx 0.5\mu b \rho^{1/2}$, where M is the Taylor factor. The magnitudes of σ_i calculated from ρ were in reasonable agreement with those obtained from the Hall-Petch plots. H-P plots gave that σ_i increased with strain ε and with decrease in temperature. This result was in accordance with Li's grain boundary ledge mechanism for the effect of grain size on the flow stress.

Chowdhury *et al.* (2005) The present paper deals with the evolution of microstructure and texture of a 316L austenitic stainless steel during cold rolling. With increasing rolling reductions, different deformation mechanisms become active. TEM observations show the presence of deformation twinning. With increasing strain the volume fraction of twin increases. At higher degrees of reduction, there is evidence that flow localization takes place and there is formation of shear bands. The evolution of texture is correlated with the associated deformation mechanisms.

Salishchev *et al.* (2006) observed the structure and properties of stainless steels subjected to severe plastic deformation. It was reported that a micro-crystalline structure forms in austenitic and austenitic-ferritic steels at transformation temperature ($t_d > 800^\circ \text{C}$) and in ferritic steel at (t_d in the range $600 - 800^\circ \text{C}$). If deformation was performed below these temperatures, the mechanism of structure formation changed, and a mixed-type of structure consisting of grains, sub-grains, and fragments, 100–300 nm in size, were formed. The results showed that steels with Sub Micro-Crystalline (SMC) structure formed by the method of SPD possessed high strength characteristics at satisfactory ductility and high corrosion resistance and damping properties. The strength of steel with SMC-structure obtained with the use of strain-induced transformation and reverse transformation was substantially higher than that after SPD.

Rajashekhra *et al.* (2007) investigated the Hall–Petch behaviour in Ultra-Fine-Grained AISI 301LN stainless steels. The yield strength obtained for all annealed samples was evaluated with respect to the two terms present in Hall–Petch relationship: the off-set strength (σ_o) and grain boundary strengthening (σ_{gbs}). The off-set strength was further subdivided into three contributions: precipitate strengthening (σ_{ppt}), solid solution strengthening (σ_{ss}), and strain hardening (σ_d), and the effect of annealing parameters on these contributions was analyzed. It was concluded that for samples annealed at 800°C , the high yield strength was primarily due to presence of ultrafine grains leading to significant grain boundary strengthening, solid solution strengthening, and secondary-phase nitrides leading to substantial precipitate strengthening. As the annealing temperature was increased to 900°C or above, the dominant strengthening mechanism to yield strength was due to solid solution strengthening. Further, strain hardening was not an important mechanism in increasing the yield strength for any of the annealed samples.

Milititsky *et al.* (2008) showed the characterization of mechanical properties of low-nickel austenitic stainless steels. The tensile properties of six low-nickel austenitic stainless steels were investigated. The Ludwigson model was shown to be adequate in describing the flow behavior of stable, low stacking fault energy austenitic alloys in the entire strain regime of a conventional tensile test. Nitrogen was found to be the most effective element in increasing the lattice friction stress, while carbon was the most effective alloying element in increasing the efficiency of grain boundary strengthening (K). The combined effect of nitrogen and

carbon resulted in high-sustained work hardening rates and a delay in the onset of dynamic softening. The addition of copper and nickel (two elements which are known to increase the stacking fault energy) had the opposite effect. The development of α -martensite also resulted in a strong increase in work hardening rate in uniaxial tension.

Misra *et al.* (2009) observed the microstructure and deformation behaviour of phase-reversion-induced for different cold deformation reductions (~45, 62, and 77 percent) and annealing temperature-time sequence (730° C to 800° C; 1 to 10 seconds) in nano-grained /ultrafine-grained type 301LN austenitic stainless steel. The phase-reversion-induced structure was observed to depend on cold rolling reduction and temperature-time annealing cycle. Austenite nucleation during phase-reversion annealing occurred in the form of thin plates or as equiaxed grains along the martensite laths. Further it was concluded that the size of reverted new austenite grains (upon annealing at 730° C for 1 to 10s) decreased with cold rolling reduction because of increased number of nuclei in deformed martensite. Further in ultrafine (~100 to 500 nm) and nanocrystalline grains (<100 to 200 nm), the formation of strain induced α – martensite was facilitated by twinning, but in submicron size grains (~500 to 1000 nm), the strain-induced nucleation of martensite was found to primarily occur at slip bands and was associated with a high density of dislocation tangles.

Kumar *et al.* (2009) have discussed the role of strain-induced martensite on microstructural evolution during annealing of metastable austenitic stainless steels. The research work mainly focused on microstructural aspects of reverted austenite in metastable austenitic stainless steel of the type AISI 304L. The material was cold rolled to 90% with and without inter-pass cooling which produced 89% and 43% of strain induced martensite respectively. The reversion of SIM to austenite took place over the temperature range of 450-800° C, when annealing treatment was given to the specimen. This reversion mechanism was diffusion controlled/shear type and showed dependence on strain induced martensite content and annealing time or annealing temperature. It was concluded that at a lower temperature, a relatively longer annealing time was taken for complete reversion of SIM as compared to a higher temperature.

Mythili *et al.* (2009) presented a study of strain induced martensite formation in a titanium modified 316 stainless steel bellow by transmission electron microscopy. The bellow was a

part of bellows sealed valve used in a sodium circuit operated in the temperature range of 150-450° C for nearly 40,000 hours. Titanium was added to austenitic stainless steel to overcome the problem of inter-granular corrosion due to sensitization. However, the addition of titanium, a strong ferrite stabilizer influenced the stability of gamma phase especially under strained conditions which showed the presence of strain induced martensite in an austenite matrix. The martensite formed was stable under the operating conditions. Long time exposure of strain induced martensite at lower temperatures (less than 450° C) lead to precipitation of fine, inter and intragranular chromium rich $M_{23}C_6$ which lead to sensitization. Hence, the addition of Ti a sensitization in austenitic steels since TiC prevents the formation of $M_{23}C_6$ thereby maintaining the Cr level in the matrix and the grain boundaries.

Kumar et al. (2009) studied the effect of cyclic thermal process on ultrafine grain formation in AISI 304L Austenitic Stainless Steels. The application of cyclic thermal process resulted in accelerated kinetics of recrystallization by imposing strain heterogeneity in steels in which austenite is more stable due to higher nickel contents. By virtue of higher austenite stability, these type of stainless steels exhibit delayed or limited α - martensite phase formation compared to metastable stainless steels of type 301 SS. As reported earlier the number of thermal cycles required for complete reversion was higher at lower temperature compared to that at high temperature. After the first cycle at 775° C, the onset of austenite nano-grain formation within the highly deformed regions could be noted. This also suggested that deformation bands and highly dislocated regions were the preferential nucleation sites for formation of ultrafine grains of austenite. The fraction of recrystallization, an average of 28 percent, was further increased after the second cycle. After the third thermal cycle, the heavily deformed structure was largely consumed by the recrystallized region and after the fourth thermal cycle, selective growth of the grains could be noted, but still the largest grain size was about 500 nm. On the other hand a two-step cyclic thermal processes, first at high and next at low temperature, produced very closely distributed ultrafine grains. Thermal cycling at 950° C produced grains beyond ultrafine grain size range ($d < 500$ nm), while the same at 925° C resulted in lowest average grain size. Overall, it was possible to obtain ultrafine grained austenite microstructure by varying temperature and number of cycles. The tensile testing of the nano or ultrafine grained austenitic stainless steel, thus processed, indicated high tensile

properties but rather poor ductility. The poor ductility was mainly imparted by the poor strain hardenability of the material.

Karjalainen *et al.* (2009) reported on the enhanced mechanical properties through reversion in metastable austenitic stainless steels. When martensite transforms into austenite by the diffusional mechanism in AISI 301LN steel, a martensitic shear-type reversion operates in AISI 301 steel as well as in 17Cr-7Ni-type experimental steels. This means that ultrafine austenite grains are formed directly by the nucleation and growth process from cold-rolled martensite in 301LN, but in the other steels they form by recrystallization from the shear-reverted austenite, thus elucidating the basic difference in the kinetics of formation of ultrafine grains. However, regardless of the reversion mechanism and subsequent microstructure, controlled reversion seems to lead to good mechanical properties both in 301LN and 301 steel. The high performance of 301LN is attributed to the microstructure consisting of ultrafine austenite, deformed austenite, and tempered martensite, and in 301 to shear reverted austenite having adequate dislocation density that leads to gradual strain hardening.

Ultrafine austenite grain size enhances the strain induced martensite formation in tensile straining. Otherwise, a high dislocation density of austenite (in 301 steel, for example) inherited through shear reversion also increases the instability of austenite and enhances the martensite formation, due to the increased flow stress of these steels during tensile straining. High dislocation density is expected to lead to enhanced strain-induced martensite formation at lower deformation strains, i.e., affecting the stability of the austenite and strain hardening rate.

Hedstro *et al.* (2009) used high-energy X-ray diffraction and material modelling to investigate the strain-rate dependence of strain-induced martensitic transformation and stress partitioning between austenite and α -martensite in a metastable austenitic stainless steel during tensile loading. It was observed that strain-rate sensitivity is high. Even moderate strain rates of 10^{-2} s^{-1} suppressed α -martensite transformation due to adiabatic heating of samples. Increasing the strain rate caused a significant decrease in α - martensite volume fraction from 0.5 (strain rate 10^{-3} s^{-1}) to 0.25 (strain rate 10^{-2} s^{-1}) at fracture. During loading,

austenite was seen to transfer the load to α -martensite as it formed and thus α -martensite carried more than twice the stress, compared to austenite.

Rajasekhara *et al.* (2009) discussed the phase and microstructure evolution of a heavily cold-rolled AISI 301LN stainless steel (SS), before and after annealing. The steel was cold-rolled to 63% rolling reduction and subsequently annealed from 600 to 1000° C for short annealing durations (1–100s). The microstructure consisted of regions of dislocation cell-type martensite (which characterized by the presence of dislocation walls and forests) and regions of heavily deformed lath-type martensite. The martensite to austenite reversion upon annealing at 600° C for 1 and 10s is negligible, but nano-scale austenite grains were formed in the martensitic matrix. Partial reversion to nano/submicron austenite grains was observed for samples annealed at 600° C for 100s, and 700° C for 1s. Samples annealed at higher temperatures exhibited a complete reversion to submicron/nano-austenite grains with a large grain size variation, as well as secondary phase chromium nitride precipitates. Austenite nucleation from martensite was sequential where dislocation cell-type martensite is consumed first, followed by austenite nucleation on geometrically necessary grain boundaries of heavily deformed lath-type martensite.

Misra *et al.* (2010) describe the electron microscopy study of microstructural evolution associated with martensitic shear phase reversion-induced nano-grained/ultrafine-grained (NG/UFG) structure in an experimental Fe–16Cr–10Ni alloy with very low interstitial content (carbon and nitrogen). The phase reversion in Fe–16Cr–10Ni occurred by shear mechanism, which is similar to that observed for AISI 301, but is different from the diffusional mechanism in 301LN steel. A relative comparison with the previous work on commercial austenitic stainless steels interestingly indicated that the diffusional reversion in 301LN occurred in the temperature range of 730–800° C, shear reversion in 301 steel occurred in the temperature range of 700–800° C, while in the experimental Fe–16Cr–10Ni phase reversion occurred in the high temperature range of 800–900° C. While the investigated Fe–16Cr–10Ni alloy and previously studied type 301 austenitic stainless steels, there were significant differences in reversion mechanism. First, in 301 steel, there was break-up of individual martensite grains that was absent in Fe–16Cr–10Ni alloy. Second, during phase reversion annealing of 301, there was evidence of individual martensite grains to produce several regions of misoriented austenite grains, whereas in the experimental Fe–16Cr–10Ni alloy,

reversed defect-free austenite grains appeared to have nearly same crystallographic orientation. Third, the reversion process occurred at 100° C greater in the Fe–16Cr–10Ni alloy in comparison to AISI 301 austenitic stainless steel.

Karjalainen *et al.* (2010) investigated the metastable type 301 austenitic stainless steels and observed that severe cold rolling (CR) of ~52 percent and controlled phase-reversion annealing in the temperature range of 700° C to 800° C by shear reversion mechanism lead to NG/UFG structure with grain size in the range of 200 to 500 nm. The reversion process involved the following stages: (a) transformation of strain-induced martensite to lath-type austenite grains with high dislocation density, (b) formation of dislocation-cell type and transformation to recovered austenite with defect-free sub-grains, and (c) coalescence of sub-grains to form NG/UFG structure. It was a clear indication of shear-type reversion mechanism of martensite to austenite and not a diffusional reversion mechanism, as observed for 301LN austenite stainless steel in which, austenite nucleates at martensite lath boundaries. At a constant annealing temperature-time condition, the yield strength was higher and corresponding elongation lower for 301 stainless steels as compared to 301LN stainless steel. The NG/UFG structure thus produced was characterized by high strength (~1000 MPa) and high ductility (~30 percent) combination.

Etienne *et al.* (2010) presented a comparative study on ultrafine-grained 316 and 304 austenitic stainless steel samples which were produced by high pressure torsion at room temperature. Their microstructure, after deformation and annealing at a temperature in the 350-900° C range, was characterized using several techniques (transmission electron microscopy, X-ray diffraction, etc.). The average grain size in the ultrafine-grained 316 was about 40nm while it was larger in the ultrafine-grained 304 due to a smaller deformation. Results showed the formation of α' - martensite during deformation in both steels while ϵ -martensite was formed only in the 304 steel. Annealing at 350° C led to decrease of α' -martensite content in 316 steel. The trend is different in the 304 steel, in which the α' -martensite content increases. This is attributed to the transformation of ϵ -martensite in α' -martensite.

Kawasaki M. (2010) reported the most promising methods of SPD i.e. Equal Channel Angular Pressing (ECAP) and High-Pressure Torsion (HPT) for refining the grain size of

polycrystalline metals to the sub-micrometer or even the nano-meter level. The work had a strong emphasis on principles of grain refinement and development of mechanical properties at high temperatures in materials after processing by ECAP; and hardness homogeneity and microstructural evolution in materials processed by HPT. Processing by ECAP provided capability of introducing excellent grain refinement and produced ultrafine grains in polycrystalline solids. Processing by HPT was also an attractive technique to produce polycrystalline materials with grain sizes typically in the nano-meter range. A gradual evolution in micro-hardness homogeneity occurred with increase in the numbers of revolutions in HPT.

Misra *et al.* (2010) studied the effect of nitrogen addition on reversion transformation of strain-induced martensite to austenite and tensile behaviour of Cr-Ni-N steel. The behaviour was compared with that of a Cr-Ni steel with extremely low levels of interstitial elements viz carbon and nitrogen. Consistent with the high metastability of investigated Cr-Ni-N steel, only 40% cold rolling reduction was adequate to transform 100% austenite to martensite, while Cr-Ni steel required ~73% reduction for complete transformation. On annealing in the temperature range of 800-900° C for 1-100s, the phase reversion of martensite to austenite occurred by shear reversion mechanism. In spite of the similarity in phase reversion mechanism and grain structure between the two steels, the Cr-Ni-N steel exhibited extensive strain hardening ability during tensile straining, consistent with its higher metastability index. The yield strength of Cr-Ni-N steel was significantly higher because of significantly higher C and N contents and correspondingly lowers elongation. Too high metastability was the reason for low elongation in this steel.

Rajasekhara and Ferreira (2011) applied a generalized phase transformation kinetics model to understand martensite to austenite transformation in cold-rolled and annealed metastable austenitic stainless steel, in order to achieve ultrafine-grained austenite. In particular, it was found that nitrogen-diffusion is responsible for rapid martensite to austenite transformation, while the process of heavy cold-rolling provides heterogeneous nucleation sites for the formation of austenite. In this regard, the geometry of austenite/martensite grain junctions is a significant parameter for promoting fast kinetics. The results indicate that martensite to austenite transformation appears to occur at austenite/martensite triple point junctions.

Ahmed and Hartmaier (2011) discussed the mechanism of grain boundary softening and strain-rate sensitivity in deformation of ultrafine-grained metals at high temperatures. Besides the conventional plastic deformation by dislocation glide within the grains, grain boundary (GB)-mediated deformation and recovery mechanisms based on absorption of dislocations into GBs were also considered. The material was modeled as an elastic continuum that contains a defect microstructure consisting of pre-existing dislocation population, dislocation sources and GBs. The mechanical response of material to an external load was calculated with this model over a wide range of temperatures. It was found that at low homologous temperatures, the model material behaves in agreement with the classical Hall–Petch law. At high homologous temperatures, however, a pronounced GB softening and, moreover, a high strain-rate sensitivity of the model material was found. Thus, it was concluded that dynamic recovery processes at GBs and GB diffusion are the rate-limiting processes during plastic deformation of UFG metals.

Shintani and Murata (2011) evaluated the dislocation density in cold rolled austenitic stainless steel by profile analysis of X-ray diffraction. It was observed that mechanical properties of austenitic stainless steel change considerably on cold rolling at room temperature because of the formation of strain-induced martensite (α') phase and work hardening induced by an increase in dislocation density in the austenitic phase. The strengthening mechanism in cold rolled steel changes with differences in dislocation density between austenitic and strain induced martensitic phase. When the difference is large mechanical properties of the steel depends on the volume fraction of α' phase. When the difference is small, the properties do not depend on volume fraction of the α' phase but instead depend on dislocation density in the austenitic phase.

2.11 Gaps in the Existing Approaches

The extensive review of literature carried out for the present study reveals that a lot of work has been reported to enhance the properties of austenitic stainless steels through grain refinement by various means. The work carried out by different researchers can be categorised into the following broad classes:

- A large number of investigators have studied the various mechanisms (Severe Plastic Deformation, Thermo-mechanical etc.) for achieving grain refinement up to micro/nano levels in austenitic stainless steels.
- Some researchers have studied the processes involving formation of strain induced martensite and the various factors influencing it. Some of the literature reported in this category also investigates the grain refinement process in presence of strained induced martensite (SIM).
- Very limited amount of work has been reported which explains the factors affecting kinetics of recrystallization (grain refining process) in the absence of SIM.
- Some researchers have co-related the grain refining process with mechanical properties achieved (mainly strength and ductility) in austenitic stainless steels.

The review of literature presents the following gaps which need to be filled:

- i. Very limited literature is available on the use of thermal cycling techniques as means to achieve grain refinement in austenitic stainless steels.
- ii. Very less literature has been reported on the kinetics of recrystallization in the absence of strain induced martensite in stable austenitic stainless steels.
- iii. Only a very few of researchers have reported on nucleation and grain growth phenomenon during thermal cycling process.

The proposed research work is an attempt to overcome the gaps listed above. In the present study, thermal cycling technique would be utilized for grain refinement in stable austenitic stainless steels. The work will help in understanding the microstructural changes taking place during thermal cycling and also the kinetics involved in the recrystallization process. Finally, the effect of various factors influencing grain refining process during thermal cycling treatment would also be investigated.

CHAPTER 3

DESIGN OF THE STUDY

3.1 Introduction

This chapter presents overall design of the study which includes methodology adopted for carrying out the research work, experimental procedure and the detail of work done in each phase of research. The objective of study, tool and techniques used to meet the objective function, selection of input variables have also been covered here.

3.2 Methodology

The methodology involves identification of controllable and uncontrollable parameters and establishment of a series of experiments to find out the optimum combination of parameters which have greatest influence on the objective function and least variation from the designed target. The study involves selection of input parameter in the process of forming ultrafine/nano grains and determining the contribution of each parameter. The work has been carried out in three main phases.

Phase-I: Preliminary phase aimed at study of recrystallization and grain growth phenomenon and the effect of various controlling factors on the process. All the analytical calculations such as dislocation density, recrystallization fraction calculations, have been done in this phase. The result of preliminary study has been used to identify the appropriate range of working temperature and holding time during the process.

Phase-II: Detailed study of the controlling parameters of the process. This includes experimental details, set-up information, operating range of process parameters, description of kinetics involved and results drawn from experiments. For selecting various process parameters, a detailed study of each parameter from the previous researchers has been done.

Phase-III: Analysis of all theoretical calculated values with experimental data and validation of result. Finally, the main conclusions to be drawn from the present research work are discussed.

3.3 Establishment of Objective Function

The objective of the present research work is to study and understand the effect of cold deformation and repetitive isothermal annealing (thermal cycling process) on the microstructure and mechanical properties of austenitic stainless steels. This also enables examining the recrystallization behavior of stable austenitic stainless steel during thermal cycling process. The proposed research in an attempt to address the following:

- To propose a novel route for processing of austenitic stainless steel for the formation of ultrafine grained microstructure.
- To study the mechanism of grain refinement upto the nano level during thermal cycling process.
- To study the effect of microstructure refinement on enhancement of properties.

The key issues to be taken up during the research work are as follows:

- To study the mechanisms of producing Ultrafine / Nano grained microstructure.
- Kinetics of recrystallization for stable austenitic stainless steels.
- Evaluation of microstructures obtained during different processes.
- Optical and Transmission Electron Microstructural (TEM) studies.

3.4 Selection of Material

As received hot rolled austenitic stainless steel plates of commercial AISI 316L grade of suitable chemical composition (shown in Table 3.1), solution treated at 1060°C for one hour and quenched in water to achieve chemical homogeneity have been taken.

Table 3.1: Chemical Composition of AISI 316L Stainless Steel

Element	Symbol	% Weight
Carbon	C	0.025
Silicon	Si	0.3
Manganese	Mn	1.2
Phosphorous	P	0.03
Chromium	Cr	16.90
Nickel	Ni	10.60
Molybdenum	Mo	2.06

3.5 Experimental Procedure

The experimental work involves the following main steps:

Step1: Cold Rolling

Austenitic stainless steels specimens were taken and cold rolled for different thickness reductions. In the present work, the different cases of thickness reduction were considered viz. from 20 to 90 percent in the steps of 10 percent reduction.

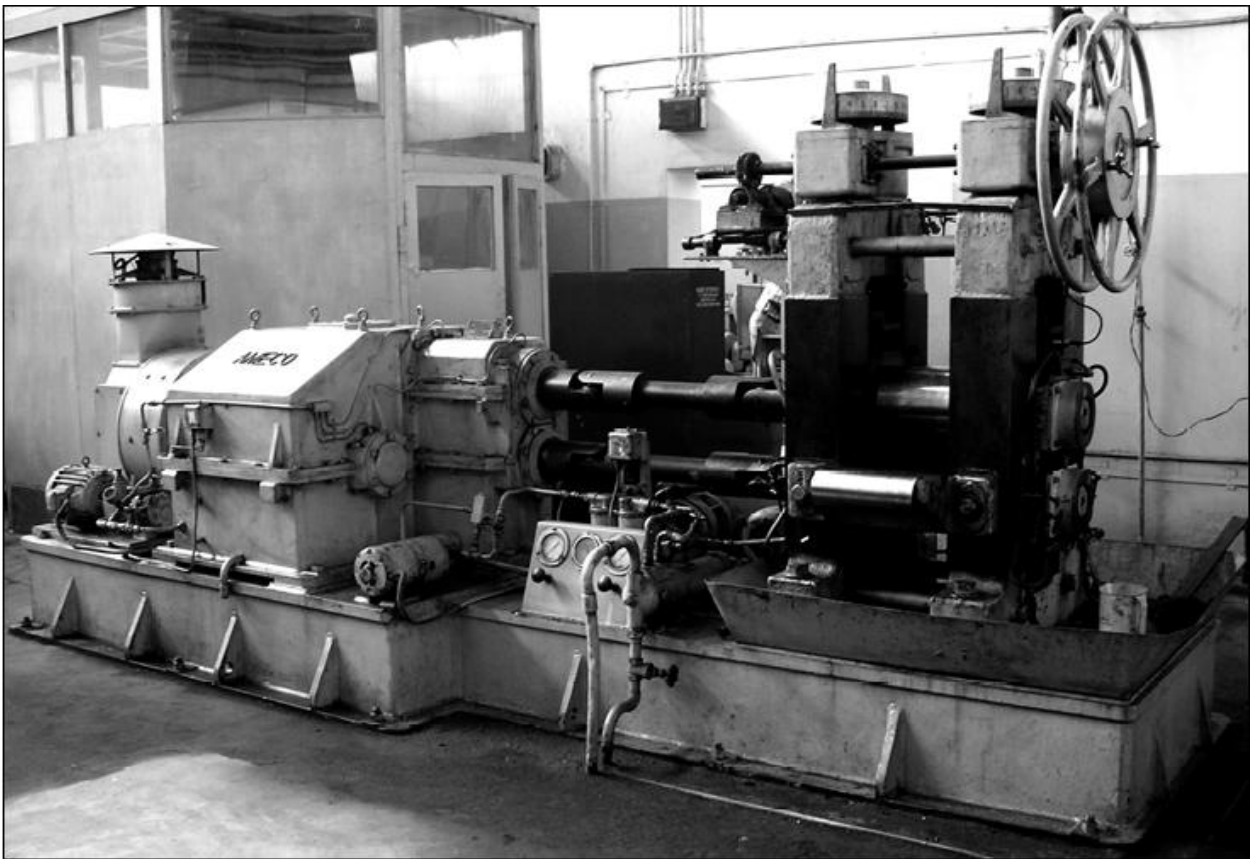


Figure 3.1 Rolling Mill for Thickness Reduction

Multi pass unidirectional cold rolling with same thickness reduction per pass has been performed in a two-high rolling mill under oil lubrication. Cold reduction was carried out with inter-pass cooling. Inter-pass cooling was performed by quenching the specimen in cold water kept at room temperature after each pass. The setup of two-high rolling mill for thickness reduction is shown in Figure 3.1.

Step2: Thermal Cycling

The cold rolled specimens of size 1cm x 1cm were subjected to cyclic thermal process at various temperatures in a muffle furnace. The shortest time taken to attain uniform temperature throughout the thickness of the specimen was determined from the model proposed by Gao *et al.*, 2000. The heating rate to be used in the present work was approximately 58°C/s. A holding time which is slightly larger than the estimated time was selected for cycle annealing. This was to ensure that equilibrium temperature reaches throughout the specimen thickness. During the heating cycle, the specimen was charged into the furnace after the desired temperature was achieved. The cooling cycle consisted of water quenching after the isothermal holding time. Thus the thermal cycle comprised of heating cycle, isothermal annealing and finally quenching in water.

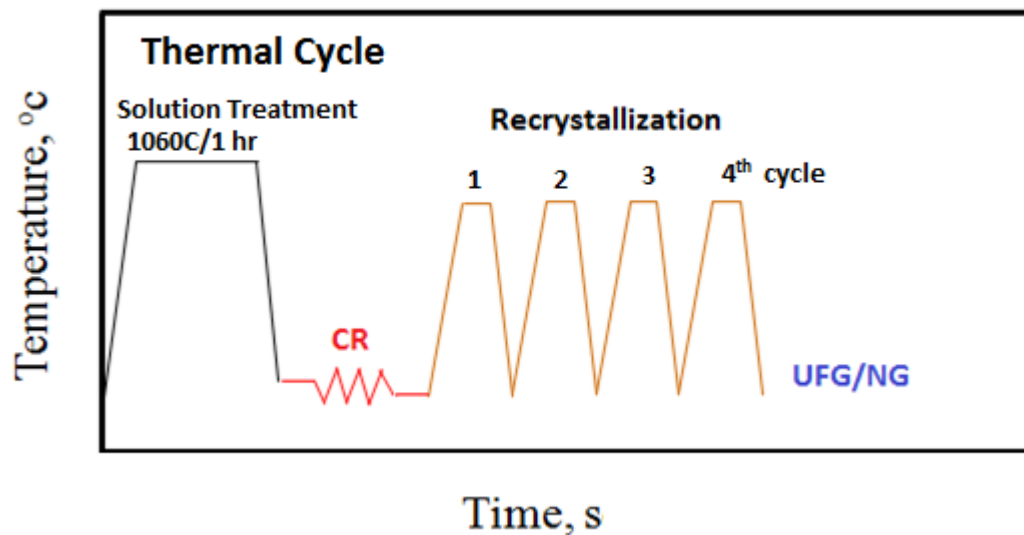


Figure 3.2 Thermal Cycling Process

Step 3: Material Characterization

After performing the thermal cycles, the material was characterized for microstructure and phase transformation. Standard metallographic techniques have been employed for the preparation of specimens for optical microscopy. To reveal the microstructure, specimens were etched with a solution of concentrated HCl (9 ml) - concentrated HNO₃ (3 ml) and CH₃OH (4 ml). The specimens for phase identification were prepared by standard metallographic techniques and electro-polishing using a bath of acetic acid and 10 percent

perchloric acid. Phase identification of electropolished specimen was performed by X-ray diffraction technique. A minimum of 10 measurements were made for each specimen to verify the reproducibility of results. A scanning electron microscope (SEM) has been used to examine the microstructural changes.

Step 4: Grain Size Measurement

The microstructures obtained through, transmission electron microscopy or optical microscopy was used to determine the average grain size. Microstructures were analyzed for recrystallization fraction and grain size determination by the *point count method* and *linear intercept method*, respectively.

Step 5: Tensile Property Evaluation

Flat tensile dogbone-shaped specimens of 25 mm gauge length as per ASTM standard E-8M were machined from the as-received plate and from thermally cycled specimens. Tensile tests were conducted at room temperature under displacement control at a strain rate of $1.3 \times 10^{-4} \text{ sec}^{-1}$ using Instron 8862 system of 100 kN capacity. Elongation was measured by an extensometer of 25 mm gauge length. The specimen has been shown in Figure 3.3 (all dimensions in mm)

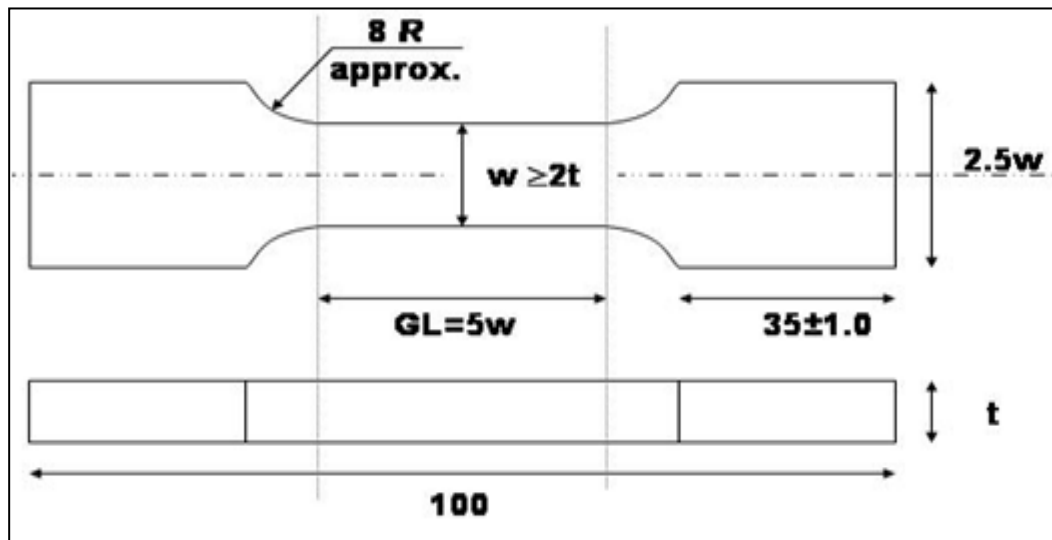


Figure 3.3: Tensile Specimen used in Study

3.6 Selection of Input Parameters

The following variables have been used as input parameters in the present work.

3.6.1 Amount of Cold Reduction

When the material is cold worked, a part of energy is stored in the material as strain energy which increases dislocation density of the material. A high dislocation density results in faster recrystallization because of high nucleation rate. The dislocation density of a material can be increased with increase in percent reduction in thickness of work material. Thus, the amount of thickness reduction is selected as an input parameter which affects the recrystallization kinetics. In the present study, initially 20 to 90 percent thickness reduction samples were used and results showed that a 90 percent thickness reduction sample stored higher amount of dislocation density in it.

3.6.2 Annealing Temperature

Annealing temperature is the working temperature for the process during thermal cycling. Thermal cycling is a temperature modulation process in which cycle is repeated at higher temperature with lesser annealing time. It is well known that at higher temperature, material recrystallize in less time while at lower temperature it takes more time to recrystallize. But at the same time, at higher temperature grain growth will takes place at faster rate as compared to lower temperature. So, the annealing temperature is selected as input parameters and should be chosen such that material will recrystallize in less time by avoiding grain growth.

3.6.3 Annealing Time

A thermal cycle consists of heating and holding of specimen followed by cooling process. Annealing time is the holding time at annealing temperature between heating and cooling process of a thermal cycle. At low annealing temperatures, a larger holding time is required to complete the recrystallization process. In the present study, a short annealing time is adopted because a small fraction of recrystallization is required after each cycle to impose strain heterogeneity between recrystallized and deformed region.

3.6.4 Number of Cycles

In order to achieve ultrafine structure in a material, the primary consideration is that nucleation rate should be high by avoiding grain growth. In this study, the material is recrystallized in a number of cycles instead of one isothermal annealing process. The number of cycles to be used depends on the fraction of recrystallization taking place in one thermal cycle. After a few thermal cycles, recrystallized fraction increases and cold deformed structure reduces in the material. In the present study, four number of cycles were considered as maximum to complete the recrystallization process.

3.7 Machines and Testing Equipment

The machines have been used for cutting, heating and cleaning of the sample and the testing equipment for determining the property of samples. The features and use of these machines and equipment has been discussed as follows:

3.7.1 Precision Cutter

A precision cutter (Make: Metkon Microcut 125 Low speed) is a compact, multipurpose, precision saw designed to cut a wide variety of materials with minimal subsurface damage as shown in Figure 3.4. The sample is mounted to a sample holder and attached to the arm. An appropriate load is applied by adjusting the counterbalancing weight and the automatic stop switch is set. The sample is positioned in any starting position relative to the diamond wheel and then a micrometer is used for precise sample positioning. With the diamond wheel rotating slowly and coolant in the reservoir, the arm is gently lowered until the sample touches the diamond wheel. Cutting continues until the automatic cutoff switch is triggered.



Figure 3.4 Low Speed Precision Cutter

3.7.2 Muffle Furnace

The muffle furnace is usually a front-loading box-type oven or kiln used for high-temperature applications. The outer body is made of mild steel sheet. The inner chamber is made of stainless steel and heaters are clamped to its outside and operated at low heat. Glass wool insulation is used between the two walls. Temperature control is provided by a capillary thermostat which possesses excellent durability and accuracy. Heaters are made of 80/20 nichrome wire, uniformly wound on the muffle. A safety thermal fuse is provided to fuse, when the temperature exceeds the set temperature. Mild steel control box is fitted on the right side of the furnace and consists of indicating lamps, On/Off switch, contactor and digital temperature controller along with Cr/Al thermocouple sensor. The furnace is electrically operated at 230 Volts (AC), Single Phase, 50 Hz supply. The furnace used in the present work is shown in Figure 3.5.

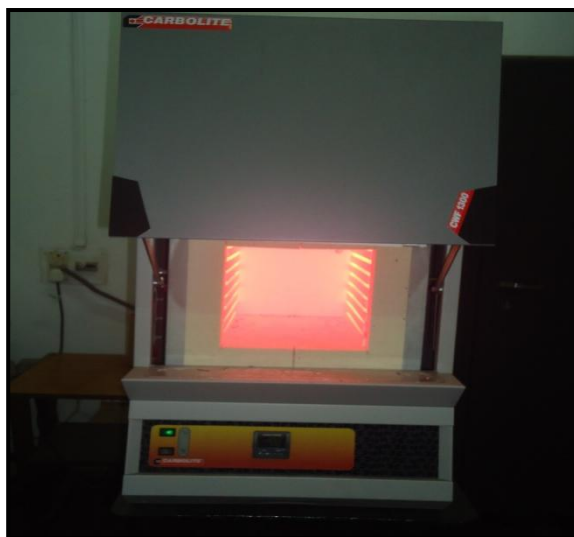


Figure 3.5 Muffule Furnace

3.7.3 Ultrasonic Cleaner

Ultrasonic cleaning uses high frequency sound waves (usually from 20–400 KHz) to agitate an appropriate cleaning solvent (sometimes ordinary tap water). Cavitation bubbles induced by the agitation act on contaminants adhering to metal surface. This action also penetrates into blind holes, cracks, and recesses. The intention is to thoroughly remove all traces of contamination tightly adhering or embedded onto solid surfaces. Water or other solvents can

be used, depending on the type of contamination and the work piece. Contaminants can include dust, dirt, oil, pigments, grease, polishing compounds, flux agents, fingerprints, soot, wax etc. In an ultrasonic cleaner, the object to be cleaned is placed in a chamber containing a suitable solution as shown in Figure 3.6.



Figure 3.6 Ultrasonic Cleaner

3.7.4 Vickers Micro Hardness Testing Machine

Micro-hardness is primarily determined with Vickers indenters under test loads in the range of 1 to 1000 gram-force and is used to measure the hardness of specific phases, small particles and brittle materials. Vickers Hardness Number is obtained by dividing the applied load in kilogram-force by the surface area of indentation. The area of the indentation produced from the Vickers square-based pyramidal diamond is determined by the mean distance between the two diagonals of the indentation.

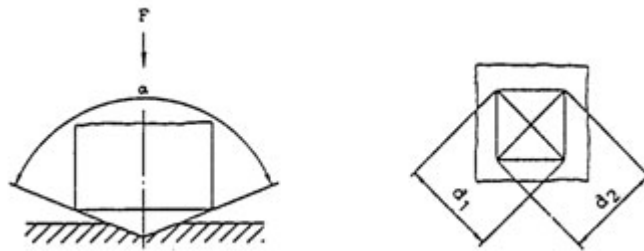


Figure 3.7 Vickers Principle

Figure 3.8 shows the Vickers Micro-hardness testing machine (Make: Leica VMHT Auto) which is used to take micro-hardness with a low load of 25 gram-force. The dwell time and indenter speed are to be taken as 15 second and 30 $\mu\text{m}/\text{sec}$ respectively.

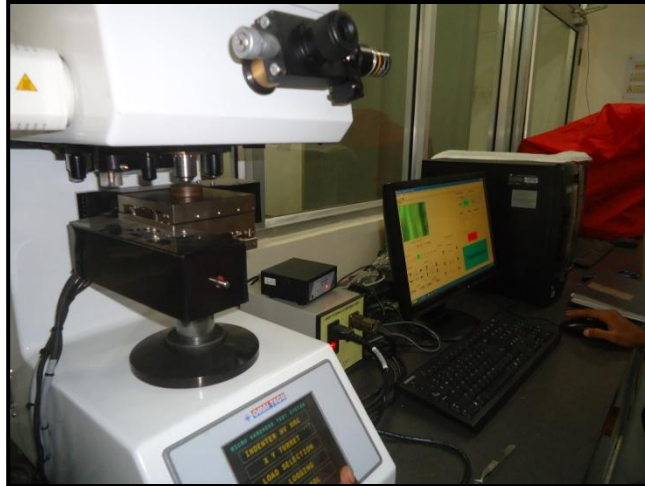


Figure 3.8 Vickers Micro-hardness Testing Machine

3.7.5 Tensile Testing Machine

The primary use of the tensile testing machine is to create the stress-strain diagram. After the diagram is generated, a pencil and straight edge or computer algorithm can be used to calculate yield strength, young's modulus, tensile strength or total elongation. For most materials, the initial resistance to force, or modulus, and the point of permanent deformation, is obtained from plots of force against elongation. The tensile testing machine used in the present work is shown in Figure 3.9.



Figure 3.9 Tensile Testing Machine

3.8 Metallographic Specimen Preparation

Metallography is the study of a materials microstructure. The basic steps for proper metallographic specimen preparation include: sectioning and cutting, mounting, planar grinding, rough polishing, final polishing, etching, microscopic analysis, and hardness testing.

3.8.1 Sectioning and Cutting

All metallographic samples need to be sectioned to the area of interest and for easier handling. Depending upon the material, the sectioning operation can be obtained by abrasive cutting (metals and metal matrix composites), diamond wafer cutting (ceramics, electronics, biomaterials, minerals), or thin sectioning with a microtome (plastics). Proper sectioning is required to minimize damage, which may alter the microstructure and produce false metallographic characterization. Proper cutting requires the correct selection of abrasive type, bonding, and size; as well as proper cutting speed, load and coolant. The machine used for sectioning and cutting is shown in Figure 3.10.



Figure 3.10 Cutting Machine

3.8.2 Mounting

Mounting of specimens is usually necessary to allow them to be handled easily. It also minimizes the amount of damage likely to be caused to the specimen itself. The mounting material used should not influence the specimen as a result of chemical reaction or mechanical stresses. It should adhere well to the specimen, and if the specimen is to be electropolished later in the preparation then the mounting material should also be electrically

conducting. Specimens can be hot mounted (about 150°C) using a mounting press either in a thermosetting plastic, e.g. phenolic resin, or a thermo-softening plastic e.g. acrylic resin. If hot mounting can alter the structure of the specimen a cold-setting resin can be used, e.g. epoxy, acrylic or polyester resin. A mounted specimen usually has a thickness of about half its diameter, to prevent rocking during grinding and polishing. The edges of the mounted specimen should also be rounded to minimize the damage to grinding and polishing discs. The mounting press and the mounted sample are presented in Figure 3.11 and 3.12 respectively.



Figure 3.11 Mounting Press with Specification

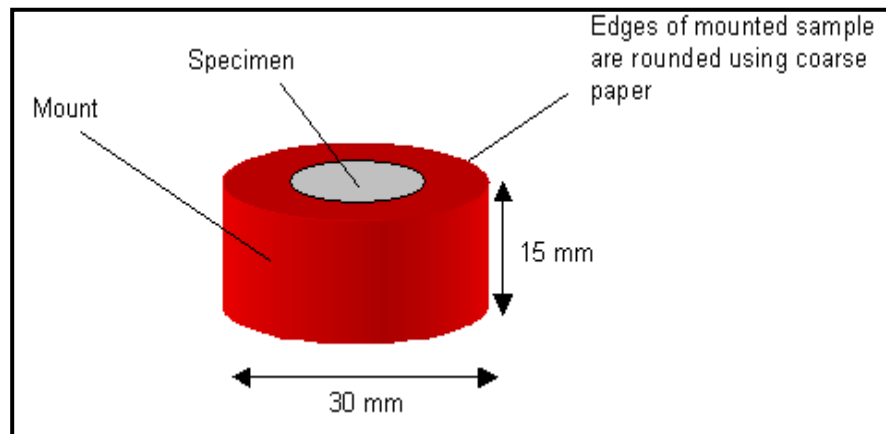


Figure 3.12 Sample Mounting

3.8.3 Grinding

Grinding is required to planarize the specimen and to reduce the damage created by sectioning. The planar grinding step is accomplished by decreasing the abrasive grit/ particle size sequentially to obtain surface finishes that are ready for polishing. The coarseness of the paper is indicated by a number: the number of grains of silicon carbide per square inch, e.g.

180 grit paper is coarser than 1200 grit. The grinding procedure involves several stages, using a finer paper (higher number) each time. Each grinding stage removes the scratches from the previous coarser paper. This can be easily achieved by orienting the specimen perpendicular to the previous scratches. Between each grade the specimen is washed thoroughly with soapy water to prevent contamination from coarser grit present on the specimen surface. Typically, the finest grade of paper used is the 2000 grit. Figure 3.13 shows the grinding machine used in the present experimental work.



Figure 3.13 Rotary Grinding Machine

3.8.4 Polishing

Polishing discs are covered with soft cloth impregnated with abrasive diamond particles and an oily lubricant or water lubricant. Particles of two different grades are used: a coarser polish - typically with diamond particles of 6 microns diameter for removing the scratches produced from the finest grinding stage, and a finer polish – typically with diamond particles of 1 micron in diameter, to produce a smooth surface. Before using a finer polishing wheel, the specimen should be washed thoroughly with warm soapy water followed by alcohol to prevent contamination of the disc. The drying can be made quicker using a hot air drier. Mechanical polishing will always leave a layer of disturbed material on the surface of the specimen. Electro-polishing or chemical polishing can be used to remove this, leaving an undisturbed surface. The polishing machine used in the present work is shown in Figure 3.14.



Figure 3.14 Polishing Machine

3.8.5 Etching

Etching is used to reveal the microstructure of the metal/alloy system through selective chemical attack. In alloys with more than one phase, etching creates contrast between different regions through differences in topography or the reflectivity of different phases. The rate of etching is affected by crystallographic orientation, so contrast is formed between grains, for example in pure metals. The reagent also preferentially etches high energy sites such as grain boundaries. This results in a surface relief that enables different crystal orientations, grain boundaries, phases and precipitates to be easily distinguished.

The specimen is etched using a reagent. For example, for etching stainless steel or copper and its alloys, a saturated aqueous solution of ferric chloride, containing a few drops of hydrochloric acid is used. This is applied using a cotton bud wiped over the surface a few of times (Care should be taken not to over-etch, however, this is a difficult point to determine). The specimen is immediately be washed in alcohol and dried. Following the etching process there may be numerous small pits present on the surface. These are etch pits caused by localized chemical attack, and in most cases they do not represent features of the microstructure. They may occur preferentially in regions of high local disorder, for example where there is a high concentration of dislocations. If the specimen is over etched, i.e., etched for too long, these pits tend to grow, and obscure the main features to be observed.

3.8.6 Leveling

Ideally, the surface to be examined optically should be perfectly flat and level. If not, then as the viewing area is moved across the surface it will pass in and out of focus. In addition, it will make it difficult to have the whole of field of view in focus - while the centre is focused, the sides will be out of focus. By using a specimen levelling press (shown in Figure 3.15) this problem can be avoided, as it presses the mounted specimen into plasticene on a microscope slide, making it levelled. A small piece of paper or cloth covers the surface of the specimen to avoid scratching.

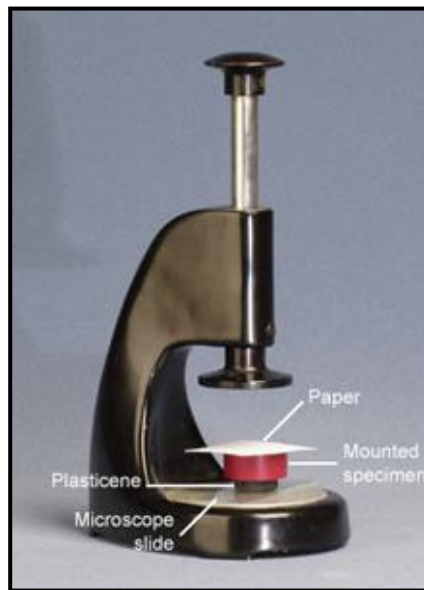


Figure 3.15 Levelling Machine

3.9 Microstructural Characterization

Knowledge of the details of formation of microstructures is essential in order to understand the relationships between processing parameters and behaviour of materials in practical application. Since the most important technological properties are strongly influenced by microstructure, this understanding is important for development of metallic materials.

3.9.1 Optical Microscopy

Metallic materials are usually opaque and therefore investigations of plane cross-sections by incident light prevail in metallography. However, the transparency of some metals and silicon to infrared light in thin sections has been effectively exploited. Optically, the individual

components of a metallic alloy differ in their amplitude and phase characteristics. While amplitude objects become visible owing to differences in light absorption and thus appear in different grey shades or even colours, phase objects only differ in the refractive indices which cannot be recognized without additional provision. The preparation of cross-sections, the enhancement of contrast by etching and other methods, as well as the microscopic set-up must be carefully optimized for the material under investigation and adjusted to the purpose of the investigation in order to get maximum information from a microscopic study. The optical microscope used in the study is shown in Figure 3.16



Figure 3.16 Optical Microscope

3.9.2 X-ray Diffraction Technique

Considering that grain size is the most important microstructural parameter to describe nanocrystalline materials, various techniques have been proposed to experimentally measure the grain size, which include transmission electron microscopy (TEM), scanning electron microscopy (SEM) and X-ray diffraction (XRD). Among these XRD has unique advantages, which includes, simple sample preparation, information from a large area etc. For nanocrystalline materials, the microstructure has been shown to be inhomogeneous with a wide distribution in grain sizes. The single parameter of average grain size appears to be insufficient to describe the microstructure features of nanocrystalline materials. The distribution range of grain size is important when considering the mechanical properties of nanocrystalline materials. In addition to grain size measurement, XRD technique has been

recently developed to measure the dislocation density and type of crystal defects in nanocrystalline metals and thereby can provide valuable information on the deformation mechanism of nanocrystalline materials.



Figure 3.17 XRD Machine

3.9.3 Electron Beam Scattered Diffraction

Electron beam scatter diffraction (EBSD) is a microstructural-crystallographic technique used to examine the crystallographic orientation of many materials, which can be used to elucidate texture or preferred orientation of any crystalline or polycrystalline material. EBSD can be used to index and identify the seven crystal systems, and as such it is applied to crystal orientation mapping, defect studies, phase identification, grain boundary and morphology studies, regional heterogeneity investigations, material discrimination and microstrain mapping etc.

EBSD is conducted using a Scanning Electron Microscope (SEM) equipped with an EBSD detector containing at least a phosphor screen, compact lens and low light CCD camera. For an EBSD measurement, a flat/polished crystalline specimen is placed into the normal position in the SEM chamber, but is highly tilted ($\sim 70^\circ$ from horizontal) towards the diffraction camera (to increase the contrast in the resultant electron backscatter diffraction pattern). The phosphor screen is located within the specimen chamber of the SEM at an angle of approximately 90° to the pole piece and is coupled to a compact lens which focuses the image from the phosphor screen onto the CCD camera.

3.9.4 Scanning Electron Microscope

A scanning electron microscope (SEM) is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample and produce signals that contain information about the sample's surface topography, composition, and other properties such as electrical conductivity. SEM can produce very high-resolution images of a sample surface, revealing details less than 1 nm in size. Due to the very narrow electron beam, SEM micrographs have a large depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This chapter deals with the overall theoretical calculations done by using some empirical formulae and presents the results of various experiments performed on the basis of calculated values. Analytical investigation has been done in order to validate the result from experimental data. In this study, an attempt has been made to understand the recrystallization behavior of heavily cold rolled AISI 316L austenitic stainless steel and its influence on grain refinement process.

4.2 Theoretical Calculations of Parameters

In the present work there are some empirical formulae have been used to calculate dislocation density in cold deformed structure and to study the kinetics of recrystallization during the annealing process. Theoretical calculation also has also been done to determine the time taken for uniform heating and heating rate for a given sample thickness.

4.2.1 Dislocation Density in Cold Deformed Material

Cold working or deformation induces dislocations in a strain free grain. The density of dislocations increases with magnitude of cold deformation. Dislocation density is considered as internal stored strain energy in the material and can be correlated with the amount cold deformation. It can be represented by an empirical equation as below (Chia *et al.*, 2005)

$$\sigma = \sigma_0 + \alpha \mu b \rho^{1/2} \quad \text{Eq. 4.1}$$

where α is a constant of the order of 0.5, σ_0 is the initial flow stress in the material and σ signifies the flow stress after cold deformation (Roucoules *et al.*, 2003)

Further, flow stress is related to strain as described by the relationship between flow stress and strain as $\sigma = K\varepsilon^n$. (*Ludwik relation*) for many materials (Samuel and Rodriguez, 2005). But several researchers have observed deviations of the flow relation from the *Ludwik relation* for stainless steels. Low and Garofalo, (1947), found that Ludwik relation was an

inadequate description of the plastic flow behavior exhibited by austenitic stainless steels and suggested that a strain induced decomposition of austenite to martensite is responsible for the exceptional behavior of this material. *Ludwigson and Berger, (1969)* presented a new stress-strain relation for metastable austenitic stainless steels (like AISI 301 steel) by using volume fractions of remained austenite and newly formed martensite. It was then understood that this deviation of the flow curve is not unique to only metals only that exhibit phase transformation. A model was later developed for annealed stable austenitic steel that does not exhibit any martenistic transformation. Then mathematical model of *Ludwigson* can be written as:

$$\sigma = K \varepsilon^n + \exp (K_1 + n_1 \varepsilon) \quad \text{Eq. 4.2}$$

Where K, K₁, n and, n₁ are constants of Ludwigson's relation. The flow curve parameters derived for austenitic stainless steel in Ludwigson's work are given as: K= 1356 MPa; n= 0.453; K₁= 5.2; n₁= 19.3 (Samuel *et al.*, 2005; Samuel and Choudhary, 2010)

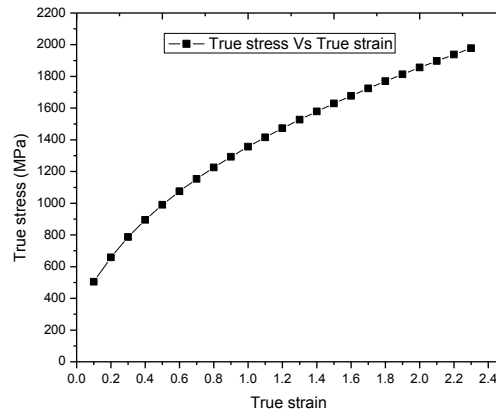


Figure 4.1 Stress - Strain Graph

By using the constant values, Figure 4.1 shows the correlation between true stress and true strain (*Ludwigson relation*). The graph does not show a linear relationship but it may vary according to work hardening relation of austenitic stainless steels.

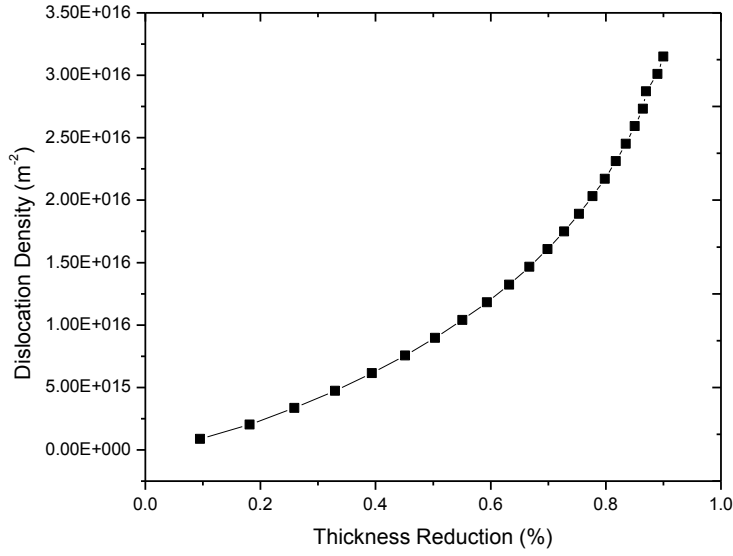


Figure 4.2 Variation of Dislocation Density

Figure 4.1 can be used to calculate dislocation density from Equation 4.1. For a given stress, dislocation density for a given increase in flow stress figure can be calculated. The values of other parameters are taken as $\sigma_0 = 210$ MPa, $\alpha = 0.5$, $\mu = 80$ GPa, $b = 2.49\text{\AA}$ (Raghavan, 2004). Figure 4.2 shows the variation of dislocation density with increase in percent of cold reduction.

4.2.2 Recrystallization Fraction during Annealing Process

The generally accepted empirical equation that best describes the isothermal kinetics of recrystallization requires measurement of the recrystallized volume fraction, X_v , and can be expressed as shown in Equation 4.3 (Kalu and Waryoba, 2007).

$$X_v = 1 - \exp [-k t^n] \quad \text{Eq. 4.3}$$

Equation 4.3 is attributed to *Kolmogorov (1937)*, *Johnson and Mehl (1939)*, and *Avrami, (1939)*, and is commonly known as JMAK. The JMAK equation is strictly valid only when the recrystallized grains are distributed randomly and when the grains are growing independently of one another. The order of the reaction n , also known as the Avrami or JMAK exponent, reflects the nucleation rate and/or the growth morphology, and k is the pre-exponential factor, a kinetic parameter depending on the annealing temperature, nucleation

rate and growth rate. In general, n takes a value in the range of $1 \leq n \leq 2$ for one-dimensional growth, $2 \leq n \leq 3$ for two-dimensional growth and $3 \leq n \leq 4$ for three-dimensional growth. For no change in mechanism, n is insensitive to temperature, whereas k is the temperature-dependent constant expressed as shown in Equation 4.4.

$$k = k_0 \exp (-Q/RT) \quad \text{Eq. 4.4}$$

where k_0 is a constant, Q the activation energy, R the gas constant ($R = 8.314472 \text{ J mol}^{-1} \text{ K}^{-1}$) and T is the absolute annealing temperature. For austenitic stainless steels, activation energy is taken as 177 KJ/mol. The value of k is calculated for different annealing temperature and pre-exponential constant is taken as 0.225×10^2 . By putting the value of k , recrystallization fraction for different annealing time viz. 60, 50, 40, 30, 20, and 10 sec corresponding to different annealing temperature is determined. The variation in amount of recrystallization fraction with respect to annealing time at different annealing temperatures is provided in Figure 4.3.

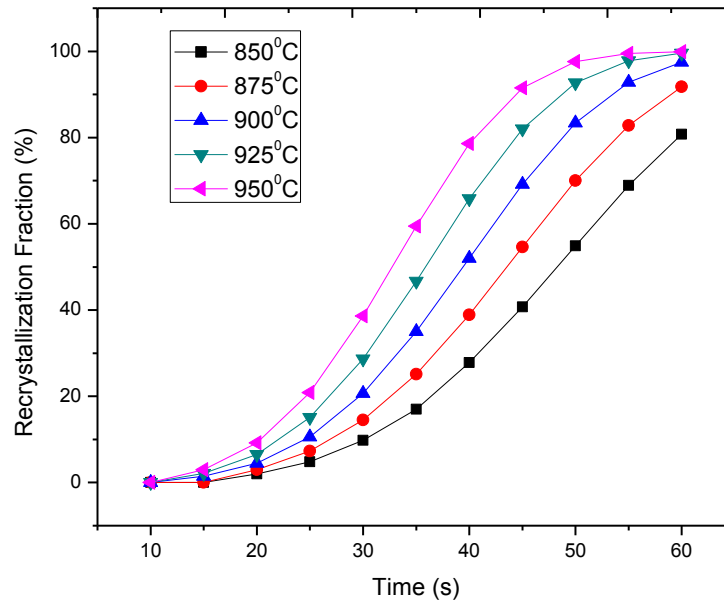


Figure 4.3 Recrystallization Fraction with Annealing Time

Figure 4.3 show that recrystallization fraction increases with increase in annealing time. A higher temperature requires less time for complete recrystallization as compared to a lower temperature.

4.2.3 Time for Uniform Heating

When a sample is heated in the furnace, a certain amount of time is required before the core of specimen reaches the furnace temperature. Initially, the surface temperature would be same as furnace temperature when the sample is put into the furnace. However, this temperature does not reach the centre of sample and takes time to become uniform throughout the cross-section of sample. Thus, a heating rate (dT/dt) is calculated by the formula (Gao *et al.*, 2000) as shown in Equation 4.5.

$$t = \frac{F_0}{\alpha} R_v^2 \quad \text{Eq. 4.5}$$

where,

$$F_0 = \frac{-\ln(\theta)}{C Bi}$$

α = thermal diffusivity = $k/\rho c$

$R_v = 3\text{Volume}/\text{Area}$

Bi = Biot Number = $h R_v/K$

h = overall heat transfer coefficient for radiant atmosphere furnace ($220 \text{ w/m}^2 \text{ }^\circ\text{C}$)

k = thermal conductivity (21.5 w/m-k)

C = constant is given as $1/(a_2 + b_2 Bi)$; $a_2 = 0.31934443$ and $b_2 = 0.099154717$

and, $\theta = (T_t - T_\infty)/(T_i - T_\infty)$

where,

T_t = temperature at the centre of sample

T_∞ = temperature of the furnace

T_i = initial temperature of the sample

From the above calculations, Gao *et al.*, 2000 calculated the heating rate as 58°C/s and time taken to reach the temperature from surface to centre as 15 seconds.

4.3 Experimental Results

After completing the theoretical calculations, several experiments were performed in order to validate the calculated values. An appropriate combination of time and temperature for thermal cycling process was found. As previously mentioned, the maximum number of

thermal cycles was restricted to maximum four. An ultrafine grained matrix was required in the structure and there was a possibility of grain growth after forth cycle. First of all, the dislocation density was calculated after each thickness reduction to find the percent thickness reduction which provides maximum energy stored. The recrystallization temperature at which a selected composition of austenitic stainless steel gets fully recrystallized was also found. Once the recrystallization temperature was determined, the recrystallization fraction was calculated at this particular temperature for different annealing time periods (holding time). These calculations have been done by using the hardness measurement of samples after each process.

4.3.1 Dislocation Density Evaluation

Austenitic stainless steel samples of AISI 316 grade were cold rolled to different thickness reductions starting from 20% to 90% in steps of 10%. Multi pass unidirectional cold rolling with same thickness reduction per pass was performed in a two-high rolling mill under oil lubrication. The macro hardness was measured by a Vicker's hardness testing machine (Reicherter Stiefel Mayer VH-3) using a load of 30 kgf for a dwell time of 5s. The average values of 10 measurements were plotted against percentage of cold reduction as shown in Figure 4.4.

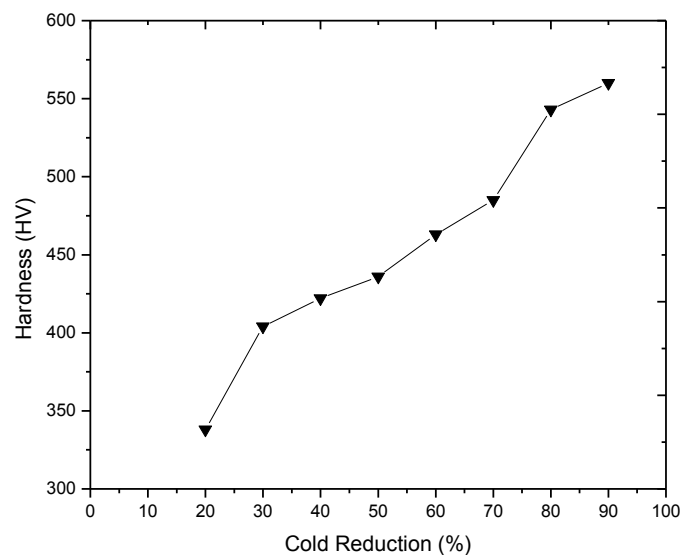


Figure 4.4 Hardness Value with Cold Reduction

It was well understood that when a material is processed under cold deformation (thickness reduction), the hardness value of the material is increased. A similar trend has been shown in Figure 4.4. This increase in hardness can be correlated with increase in dislocation density. The dislocation density increases with cold reduction and actual value of dislocation density is calculated by taking hardness values of different samples by a correlation equation called Linear Bailey-Hirsch relationship (Narutani and Takamura, 1991). The relation is shown in Equation 4.6.

$$HV [GPa] = 0.7 + 3 \times 10^{-8} \rho^{1/2} \quad \text{Eq. 4.6}$$

where,

HV = Vickers Hardness (GPa)

ρ = Dislocation density (m^{-2})

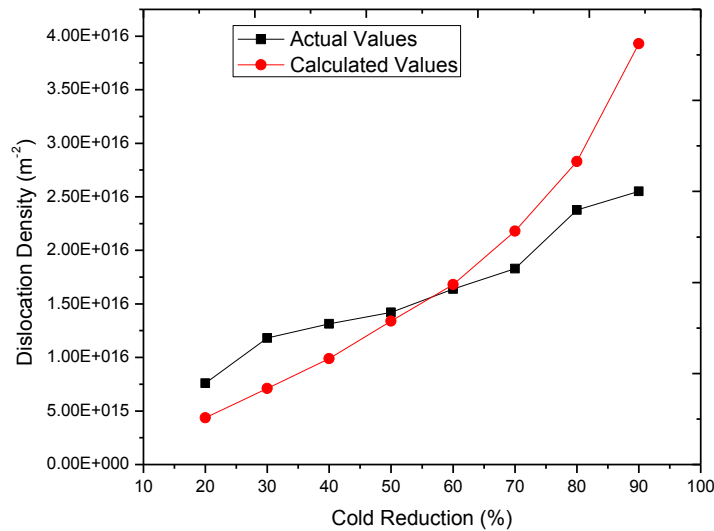
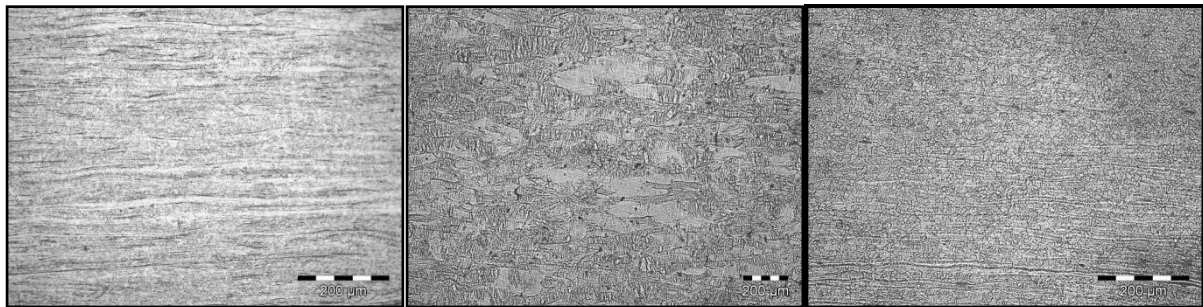


Figure 4.5 Dislocation Density vs Cold Reduction

Figure 4.5 shows the comparison of actual dislocation density values with calculated values from the empirical relation. It was observed that the slope of dislocation density increases with increase in percent of thickness reduction. For a maximum amount of dislocation density, 90 percent of thickness reduction was selected for the present study.

4.3.2 Recrystallization Temperature

There are two ways to determine the recrystallization finish temperature under different isothermal annealing conditions; one is the microstructure observation method and the other is the hardness test method. In the former case, a large number of samples are required for different annealing time periods especially in the temperature range near the recrystallization finish temperature. Muljono *et al.* (2001) used the microstructure observation method to determine recrystallization finish temperature. In the present study, the microstructure observation method has been used to determine the recrystallization finish temperature of austenitic stainless steel samples. The samples were heated from 700- 950°C in intervals of 25° C for 1 minute each. Microstructures show highly dislocated region (or deformed structure) in the temperature range of 700 - 800° C and a partial recrystallized region starts to grow by further increase in temperature. At 900° C, almost complete recrystallization takes place in the material and further increase in temperature results an increase in austenitic grain size. Figure 4.6 shows a clear difference in microstructure of un-recrystallized, partial recrystallized and fully recrystallized austenitic stainless steel.



(a) Un-recrystallized (800° C) (b) Partial-recrystallized (800 - 900° C) (c) Recrystallized (900° C)

Figure 4.6 Optical Image of 316L Austenitic Stainless Steel at Different Temperatures

4.3.3 Fraction of Recrystallization

Cold rolled specimens with 90 percent thickness reduction were subjected to annealing treatment for different temperatures and holding periods in a muffle furnace. The results showed a good relation of theoretical calculation with the actual measured value of recrystallization fraction. The fraction of recrystallization has been calculated by taking hardness values measured by Vickers Hardness tester. The following relation as shown in

Equation 4.7 has been used to determine the recrystallization fraction (X) for a given annealing time (t) (Kumar *et al.*, 2009):

$$X = \frac{H_0 - H_t}{H_0 - H_a} \quad \text{Eq. 4.7}$$

where

H_0 = Hardness of cold deformed structure

H_t = Hardness of annealed material in time (t)

H_a = Hardness of solution annealed material

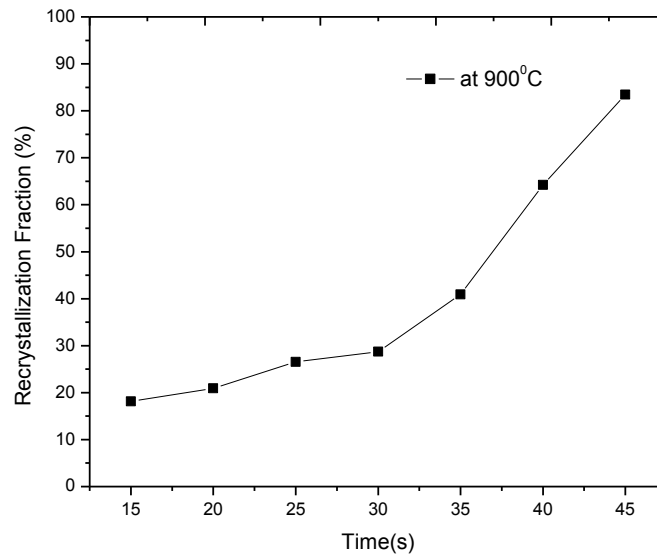


Figure 4.7 Recrystallization Fraction vs Annealing Time

Figure 4.7 shows the actual volume of recrystallized fraction in the material corresponding to different annealing periods at a constant annealing temperature of 900°C. It was observed from the above figure that around 50 % of recrystallization fraction was completed in an annealing time of 35 sec. The present study involves thermal cycling process based on strain heterogeneity induced between recrystallized portion and cold deformed region. It was required to complete 50 % fraction of recrystallization in first thermal cycle. Hence, a suitable annealing time was selected as 35 second for the present study.

4.4 Factors affecting Recrystallization Kinetics

There are several factors which affect the recrystallization kinetics appreciably. These factors are discussed as follows:

4.4.1 Effect of Prior Cold Deformation on Recrystallization

It is well known that higher deformation prior to recrystallization results in faster recrystallization kinetics. This has been attributed to the increased level of stored energy at higher deformation, thus providing a greater driving force for recrystallization and also an increase nucleation rate.

4.4.2 Effect of Recovery on Recrystallization Kinetics

The extent of recovery that occurs during recrystallization is directly related to the kinetics of sub grain growth, which depends not only on the stored energy of deformation at a site but also on the evolution of misorientation between the sub grains as they grow. Hence, recovery plays a dual role in recrystallization. While it promotes nucleation by sub grain growth, it also slows down the recrystallization kinetics by reducing the driving force, especially in the early stage of recrystallization. In the present study, the role of recovery increased due to increased boundary mobility which resulted in an acceleration of recrystallization kinetics (Radhakrishnan *et al*, 1998).

4.4.3 Effect of Annealing Temperature and Time on Recrystallization

In order to study the effect of annealing on recrystallization behavior of cold deformed structure, specimens were annealed isothermally for short durations at different temperatures. The progress of recrystallization was influenced both by temperature and time. At lower annealing temperatures (800° C), cold deformed fibrous structure was stable for much longer annealing durations compared to that at higher temperatures. It was observed that even at short holding time (of one minute) at a temperature of 900° C, it was possible to get a fully recrystallized microstructure. Therefore, 900° C was considered as the suitable temperature for studying the effect of short annealing treatments in the present investigation. This is because during cyclic thermal processes, a short holding time at the peak temperature is desirable. Thus, the entire study in the present work has been conducted at this temperature.

4.5 Evaluation of Microstructure during Thermal Cycling Process

Cyclic thermal processing exhibited very encouraging results on grain refinement. Interestingly, through this processing route, it was possible to obtain ultrafine grains even at annealing temperatures as high as 900° C, avoiding or minimizing secondary recrystallization (grain growth). The accelerated behavior observed in this study was attributed to the non isothermal part of cyclic thermal processing, during heating and cooling segment.

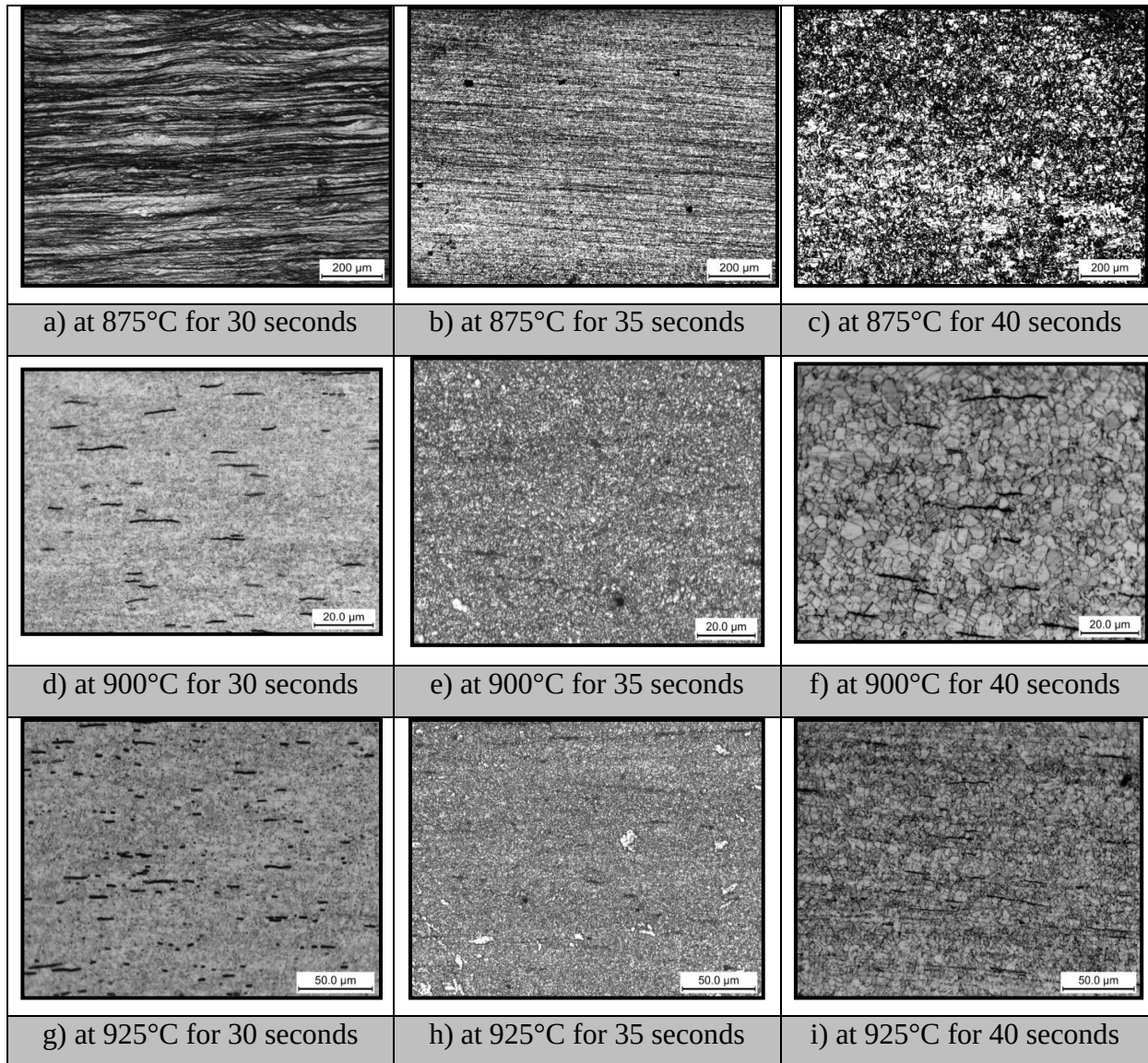


Figure 4.8 Microstructure of 316L Austenitic Stainless Steel

A review of literature reveals that recrystallization fraction is very small after the first thermal cycle. On subsequent thermal cycles, this fraction increases quite rapidly. During thermal cycling, after each thermal cycle, the strain heterogeneity increases further due to the formation of new strain free austenite grains and some volume of sub-grains which are ready to become critical nuclei (Baos and Hargreaves, 1948; Kozeschnik *et al.*, 1999). At the same, time some residual deformed region is retained in the microstructure. Thus, a non homogenous distribution of stored energy results in varying recrystallization kinetics spatially. The spatial strain heterogeneity may be the driving force for accelerated recrystallization kinetics according to the predictions made by Luo *et al.* (2004).

Thermal cycles resulted in ultrafine austenite grain formation within the highly deformed regions. The residual cold deformed microstructure continued to diminish with increasing number of cycles and an increase in the grain size also occurred simultaneously. Hence, it was found that maximum number of cycles should be restricted to four as given in literature. Increasing the number of cycles beyond this number could result in increase of grain size. As discussed earlier, the microstructure has been obtained at selected combination of temperature and time, details of which are shown in Figure 4.8.

A review of microstructures revealed that the sample having thermal cycled at 900°C for 35 s shows ultrafine grained structure as shown in Figure 4.11 and also found by grain size measurement. This microstructure shows a negligible amount of deformed region while other microstructures give an indication of either un-recrystallized region or occurrence of slight grain growth.

4.6 Determination of Initial Grain Size of Annealed Microstructure

Figure 4.9 shows the microstructure of solution annealed AISI 316L SS obtained by heating upto 1060°C for 1 hour followed by water quenching. Solution treatment is generally given to austenitic stainless steel in order to achieve chemical homogeneity. The solution treated 316L SS specimen was characterized by polygonal grains along with annealing twins. Cold rolling of solution treated specimen of polygonal grains resulted in general elongation of grains in the rolling directions. The TEM observations of cold rolled specimen with dislocation tangled and banded structure has been shown in Figure. 4.10. The estimated average grain size of solution treated sample was found in the range of 90 - 120µm.

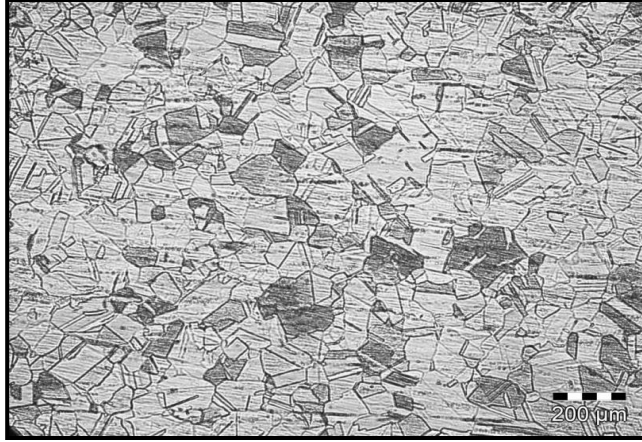


Figure 4.9 Microstructure of the Solution Annealed 316L SS

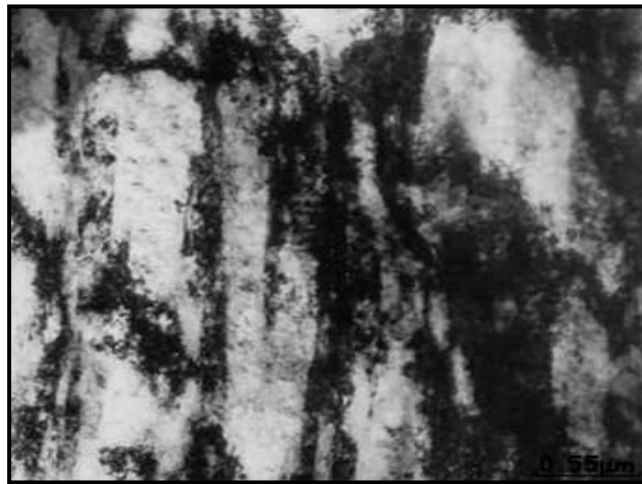


Figure 4.10 TEM Image of Cold Deformed Structure

4.7 Determination of Final Grain Size after Thermal Cycling Process

Figure 4.11 shows the microstructure of thermal cycled sample at 900° C for 35 sec in each cycle. From the above study it was found that a combination of suitable annealing temperature and time (i.e. 900° C for 35 sec) for 4 cycles gives appropriate result in terms of grain size reduction. The microstructure shows a uniform distribution of ultrafine grains ($< 1\mu\text{m}$) with a very small portion of deformed structure. The average grain size was found to be in the range, 800- 1200nm.

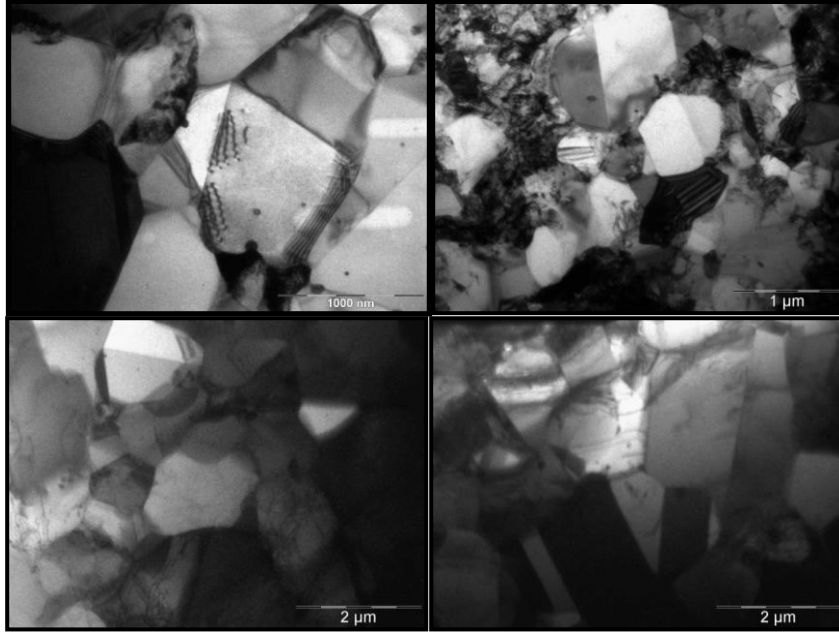


Fig.4.11 TEM Images of Ultrafine Grained Microstructure (TR 90%, 900°C, 35 Sec)

4.8 Effect of Ultrafine Microstructure on Tensile Properties

The engineering stress strain curve of solution treated and 90% cold rolled specimens is shown in the Figure 4.12.

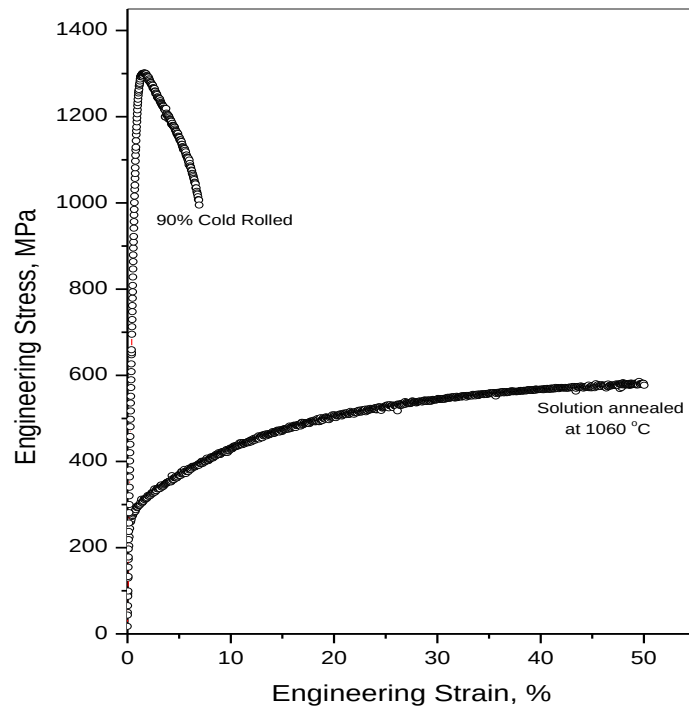


Figure 4.12 Stress Strain Curves for Solution Annealed and Cold Rolled Specimen

As expected, the solution annealed specimen showed a high ductility (~50%) with very low yield strength (268 MPa). Similarly, cold deformation led to reduction in the ductility with very high increase the yield strength (1265 MPa). This can be attributed to strain hardening of the material due to cold rolling.

The mechanical properties of the cold rolled austenitic stainless steel exhibited a very high 0.2% offset yield strength, which around six times of the solution treated coarse grained stainless steel, and a very poor total tensile elongation. The steel with high yield strength, i.e. 6 times or more, showed a total elongation as low as 1-2% as shown in Figure 4.12.

The cyclic thermal process resulted in the formation of various microstructures. This process strongly influenced the tensile properties of the material. Figure 4.13 exhibits a typical engineering stress strain curve of NG/UFG austenitic stainless steel corresponding to the microstructures presented in Fig 4.11. The yield strength was observed to be at least four times better than that of solution annealed specimen, but with only a slightly reduction in ductility. Thus, ultrafine grained microstructure exhibited a very good combination of strength and ductility.

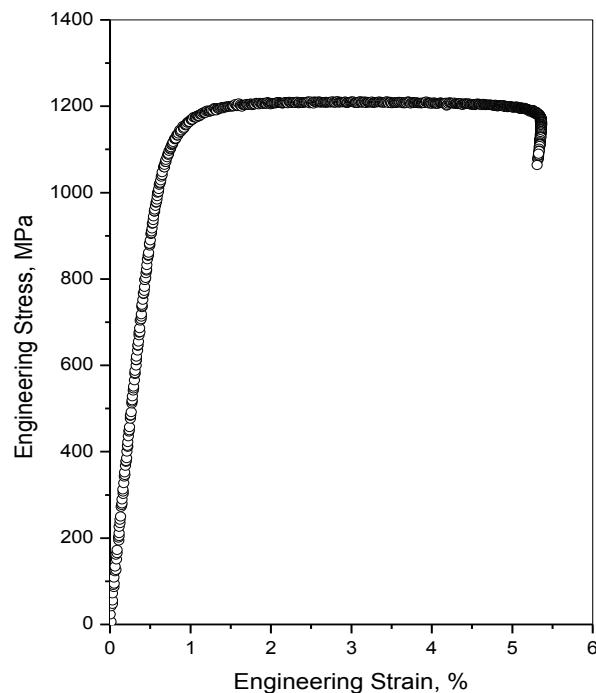


Figure 4.13 Stress- Strain Curve for Thermal Cycled Specimen

Figure 4.13 demonstrates a tensile curve with almost negligible strain hardenability in the plastic regime. The corresponding microstructure revealed a grain size of the order of 1155nm. These fine grains were noted to consist of large number of dislocations within it. The observed poor strain hardenability of this specimen could be due to the presence of dislocation in the fine grain, though not fully saturated. Probably, synergistic effects of fine grain size with large number of dislocation were responsible for poor dislocation storing capacity leading to poor strain hardenability.

4.9 Discussion of Result

The present study was undertaken to examine the possibility of grain refinement of stable austenitic stainless steel (AISI 316L). Stable austenitic stainless steel does not undergo any phase transformation unlike their metastable counterparts in which formation of strain induced martensite takes place. In the metastable austenitic stainless steels, presence of larger volume fraction of SIM promotes higher nucleation rate leading to fine grain formation (Kumar *et al.*, 2009). SIM reversion is considered to be responsible for grain refinement when present in large volume fraction but it is mainly the heterogeneity in nucleation and growth behavior that is responsible for austenite grain refinement in the absence of SIM. Further, the heterogeneity in nucleation and growth behavior can be due to spatial stored energy variation in the specimen, which is a function of cold deformation and thermal cycling. Hence, a thermal cyclic processing route has been developed for the formation of ultrafine grained AISI 316L in the absence of SIM transformation. This also describes the mechanism for grain refinement and recrystallization behavior of heavily cold rolled stable austenitic stainless steel.

Interestingly, thermal cyclic process results in different recrystallization and grain growth behavior as compared to isothermal annealing. The cyclic thermal process is found to promote grain refinement much better than the short time isothermal annealing process. This difference mainly stems from the imposition of strain heterogeneity in the microstructure after each thermal cycle (Kumar *et al.*, 2010).

In general, the recrystallization kinetics of cold deformed structure follows the JMAK (Johnson-Mehl-Avrami-Kolmogorov) type model. Krishnan *et al.* (2006) reported accelerated recrystallization and showed that it follows the JMAK type relation as given in Equation 4.8.

$$X = 1 - \exp [-(k t)^n] \quad \text{Eq. 4.8}$$

where X is the fraction recrystallized after time t and the exponent n depends on the mechanism of recrystallization. The accelerated behavior observed in this study is attributed to the non isothermal part of cyclic thermal processing, during the heating and cooling segment. This influences the constant factor k (Equation 4.8), and the other parameter, exponent n , is governed by recrystallization mechanisms. It has been assumed that influence on parameters 'k' is almost negligible, as thermal cycle has been maintained between room temperature to peak temperature, contrary to Krishnan *et al.* (2006). The heating and cooling segments have been very rapid, resulting in minimum contribution from these two factors.

Luo *et al.* (2004) studied the influence of static recrystallization on Avrami exponent for C-Mn steels. The authors observed that Avrami exponent is highly influenced by strain heterogeneity in austenite rather than in interphase boundary area. The exponent decreases with increasing strain heterogeneity resulting in increase in recrystallization kinetics (Kumar *et al.*, 2009). Hence, the main influencing factor on recrystallization kinetics could be the changed stored energy spatially after each subsequent thermal cycle. On the other hand, once the formation of austenite grains takes place, its growth rate on subsequent thermal cycles, determines the final grain size distribution. The average grain growth rate can be represented as provided by Kozeschnik *et al.* (1999) and shown in Equation 4.9.

$$G^G(t) = C_G t^{-n} \exp\left(\frac{-Q_G}{RT}\right) \quad \text{Eq. 4.9}$$

where, C_G is a constant, Q_G is the activation energy of interface migration, n is an exponent associated with decrease of driving force with annealing time (i.e. the stored energy), R is the gas constant and T is the annealing temperature. In order to attain a uniform or controlled average grain size and its distribution, it is necessary to accelerate the nucleation rate and retard the grain growth. The driving force for recrystallization is lattice defect energy, mainly dislocation/ vacancies where as for grain growth it is only grain boundary energy as it occurs by grain boundary migration. The migration of grain boundary is defined as the displacement of boundary perpendicular to the tangent plane and is often expressed as shown in Equation 4.10 (Chen *et al.*, 2000).

$$V = MP \quad \text{Eq. 4.10}$$

Where, M is the boundary mobility and P is the driving force. The rate of migration has been observed to be dependent on the misorientation, boundary inclination, temperature and impurities. After the first thermal cycle, microstructure showed a small recrystallized strain free region with high dislocation density in the cold deformed structure. It has been found that the driving force for nucleation in the deformed structure is more than the driving force required for grain growth in recrystallized region. This may possibly be attributed to a high nucleation rate after each thermal cycle which results in grain refinement.

CHAPTER 5

CONCLUSIONS

5.1 General

The development of high strength steels characterized by high strength–high ductility combination is a subject of significant interest worldwide. The demand for high strength stainless steels with characteristic properties of good strength, high ductility, toughness, workability and corrosion resistance is expected to increase in future. There is a continued and significant interest to develop high strength steels without compromising on ductility and toughness. In this regard, grain size reduction and refinement of microstructure dominate the effort, with strong emphasis on thermo-mechanically controlled processing (TMCP).

Research on cold rolling strengthening of austenitic stainless steels has taken place over the last two decades and the mechanism and phenomena are quite well understood. Also strength grades have been standardized for industrial use and design. However, anisotropy and asymmetry in mechanical properties present in these grades still call for further research to be accounted for. Isothermal annealing for grain size refinement of austenitic stainless steels, reported more than fifteen years ago, is once more under increasing interest and may result in certain industrial applications. A route for improving the strength of austenitic stainless steels without degrading their ductility is to thermal cycle heavily cold-rolled specimens to produce nano/submicron austenite grain structures. An understanding of this two-step process, (thermal cycling process) which involves cold-rolling and repetitive annealing is the key to development of some high strength austenitic stainless steels. More recently, several studies have demonstrated the formation of ultra-fine austenite grains in metastable austenitic Cr–Ni steels, while others have demonstrated this concept in commercial metastable austenitic AISI 301 and AISI 304 steel grades. The development of ultra-fine grain structures in stable austenitic stainless steel grades (AISI 316L) is also important because it opens up the possibility of using high strength austenitic stainless steel in structural applications. In the present study, considerable research has been conducted to understand phase evolution, microstructure, and mechanical properties of heavily cold-rolled and annealed AISI 316L steel grade.

The present research work was an attempt to study and understand the mechanism of producing ultra-fine or nano grained microstructure in AISI 316L austenitic stainless steels. The research also studied the effect of cold deformation and thermal cycling on microstructure and mechanical properties of austenitic stainless steels.

5.2 Results and Conclusions

The main results and conclusions to be drawn from the present work are as follows:

- AISI 316L stainless steels contain thermodynamically stable austenite at room temperature, which cannot be transformed to strain-induced martensite by deformation below the martensite formation (M_d) temperature. The XRD analysis revealed that a negligible amount of SIM is present in this grade of stainless steels.
- The flow stress of austenitic stainless steel increased with increase in true strain as per Ludwigson relation. The flow stress was calculated as 1800 MPa for 90 percent cold reduction.
- The dislocation density of AISI 316L stainless steel with 90 percent cold deformation was calculated to be $4.2 \times 10^{16} \text{ (m}^{-2}\text{)}$ which was comparable to the actual value of $2.5 \times 10^{16} \text{ (m}^{-2}\text{)}$ calculated through hardness measurements. The discrepancy could be due to the statistical model used for calculation. An increase in dislocation density takes place due to creation of new dislocations and accumulation of existing dislocations with increase in amount of plastic deformation.
- The increase in Vickers Hardness values from 350 HV to 560 HV with cold deformation from 20 to 90 percent thickness reduction was due to the energy stored in material and an increase in the dislocation density.
- The recrystallization fraction corresponding to different annealing time periods viz. 15 to 45 seconds at constant annealing temperature (900°C) was found to increase non-linearly. The results showed that for an annealing period of 15s, recrystallization fraction was 20 percent; for 30s it was upto 30 percent; while for 45s it reached nearly 85 percent.
- The microstructure of the material after cold rolling and thermal cyclic process was studied by optical and transmission electron microscope which showed formation of ultrafine grains under the condition of four thermal cycles at 900°C for 35s. Further

increase in temperature and time resulted in initiation of grain growth as revealed through microstructural analysis.

- The initial grain size of solution annealed microstructure at annealing temperature of 1060°C for 1 hour was found to be in the range of 90-120 μm . However, after processing through thermal cycling with the selected process parameters (annealing temperature of 900°C; annealing time of 35s; numbers of cycles equal to four), the grain size reduced to 1200nm.
- The tensile testing of cold deformed (90 percent reduction) austenitic stainless steel showed an increase in yield strength upto 1265 MPa in comparison to the original strength of 268 MPa (for the coarse grained solution annealed material) with an appreciable reduction in ductility. However, for cold deformation followed by thermal cycling of the material provided a combination of high yield strength (1100 MPa) and good ductility.

5.3 Major Conclusions and Recommendations

- Attempts to find ultrafine grains in the thermal cycle processed microstructure are successful. Optical microscopy revealed various microstructures with deformation band, annealing twins, and polygonal grains.
- The retained austenite grains which are considered difficult for grain refinement by the conventional thermo-mechanical processing, were observed to undergo grain fragmentation through evolution of sub-structure or sub-grain formation. The formation of sub-structure or increase in grain orientation spread may be as a result of cooling segment of the thermal cycle.
- Thermal cycling appears to have retarded the grain growth kinetics and increased the recrystallization kinetics by imposing strain heterogeneity between recrystallized and deformed regions.
- Fine grained materials exhibit a lower recrystallization temperature at low stored energies than their coarse grained counterparts. The opposite is true for higher stored energies, due to larger strains required to reach the stored energy level by a coarse grained material.

- The poor strain hardenability of nanostructure or ultrafine grained polycrystalline materials is attributed to the poor dislocation storing capacity of such grains.
- The mechanical properties of nano/ ultrafine grained austenitic stainless steels, processed by thermal cycling, indicated high yield strength with a good combination of ductility. The strength and hardness have been found to increase with decreasing grain size.

5.4 Scope of Future Work

An increasing amount of evidence of the novel properties of ultrafine/ nano grained materials has been obtained in recent times. The study can be extended to investigate the fatigue and creep behaviour of ultrafine grained/ nano structured austenitic stainless steels. This kind of study can be crucial when considering the use of nano structured materials in practical applications. Apart from the microstructural studies, analysis of texture evolution can also be further investigated after thermal cycling process. The work can also be explored to study the amount of thermal stresses induced in the material during quenching after each cycle and the effect of these stress on the recrystallization kinetics.

REFERENCES

- Ahmed, N. and Hartmaier, A. (2011), 'Mechanisms of Grain Boundary Softening and Strain-Rate Sensitivity in Deformation of Ultrafine-Grained Metals at High Temperatures', *Acta Materialia*, Vol. 59, pp. 4323–4334.
- Avrami, M. (1939), 'Kinetics of Phase Change – I. General Theory', *Journal of Chemical Physics*, Vol. 7, pp. 1103-1112.
- Baker, I., Liu, L. and Mandal, D. (1995), 'The Effect of Grain Size on the Stored Energy of Cold Work as a Function of Strain for Polycrystalline Nickel', *Scripta Metallurgica et Materialia*, Vol. 32, No. 2, pp. 167-171.
- Baos, W. and Hargreaves, M. E. (1948), 'On the In-homogeneity of Plastic Deformation in the Crystals of an Aggregate', *Proceedings of Royal Society of London A*, Vol. 193, Issue 1032, pp. 89-97.
- Callister, W.D. (1997), 'Materials Science and Engineering', *John Wiley & Sons Inc.*, 5th ed., pp. 167-168.
- Chen, S.P., Todd, I. and Vanderzwaag, S. (2002), 'Modelling the Kinetics of Grain Boundary Nucleated Recrystallization Processes after Cold Deformation', *ASM- TDS-Springer*, Vol. 33A, pp. 529-537.
- Chia, K.H., Jung, K. and Conrad, H. (2005), 'Dislocation Density Model for the Effect of Grain Size on the Flow Stress of a Ti–15.2 at % Mo β alloy at 4.2–650K', *Materials Science and Engineering A*, Vol. 409, pp. 32–38.
- Chowdhury, S.P., Das, S. and De, P.K. (2005), 'Cold Rolling Behaviour and Textural Evolution in AISI 316L Austenitic Stainless Steel', *Acta Materialia*, Vol. 53, pp. 3951–3959.
- Davis, J.R. (1994), 'ASM Specialty Handbook; Stainless Steels', ASM International.
- Di Schino, A., Salvatori, I. and Kenny, J. M. (2002), 'Effects of Martensite Formation and Austenite Reversion on Grain Refining of AISI 304 Stainless Steel', *Journal of Materials Science*, Vol. 37, pp. 4561-4565.
- Di Schino, A., Kenny, J.M. and Abbruzzese, G. (2002), 'Analysis of the Recrystallization and Grain Growth Processes in AISI 316 Stainless Steel', *Journal of Materials Science*, Vol. 37, pp. 5291 – 5298.
- Di Schino, A., Abbruzzese, G., and Kenny, J.M. (2003), 'Recrystallization and Grain Growth in Austenitic Stainless Steel: A Statistical Approach', *Journal of Material Science and Technology*, Vol.19, pp. 119-121.
- Eskandari, M., Kermanpur, A. and Najafizadeh A. (2009), 'Formation of Nanocrystalline Structure in 301 Stainless Steel Produced by Martensite Treatment', *Metallurgical And Materials Transactions A*, Vol. 40, pp. 2241-2248.
- Etienne, A., Radiguet, B., Genevois, C., Le Breton, J.M., Valiev, R. and Pareige, P. (2010), 'Thermal Stability of Ultrafine-Grained Austenitic Stainless Steels', *Materials Science and, Engineering A*, Vol. 527, pp. 5805–5810.
- Gao, M., Reid, C.N., Jahedi, M. and Li, Y. (2000), 'Estimating Equilibration Times and Heating/Cooling Rates in Heat Treatment of Workpieces with Arbitrary Geometry', *Journal of Material Engineering and Performance*, Vol. 9, Issue 1, pp. 62–71.

- Hedstrom, P., Lindgren, L.E., Lmer, J., Lienert, U., Bernier, J., Turner, M. and Oden M. (2009), 'Load Partitioning and Strain-Induced Martensite Formation during Tensile Loading of a Metastable Austenitic Stainless Steel', *Metallurgical and Materials Transactions A*, Vol. 40A, pp. 1039-1048.
- Humphreys, F.J. and Hatherly, M. (1996), 'Recrystallization and Related Annealing Phenomena', Oxford, UK: Pergamon, Elsevier Science Ltd, pp. 281.
- Johnson, W.A. and Mehl, R.F. (1939), *Transactions of Metallurgical Society*, A.I.M.E., Vol.1.35, pp. 416-458.
- Kalu, P.N. and Waryoba D.R. (2007), 'A JMAK-Micro Hardness Model for Quantifying the Kinetics of Restoration Mechanisms in Inhomogeneous Microstructure', *Materials Science and Engineering A*, Vol. 464, pp. 68–75.
- Karjalainen, L.P., Taulavuori, T., Sellman, M. and Kyröläinen, A. (2008), 'Some Strengthening Methods for Austenitic Stainless Steels', *Steel Research International*, Vol.79, No. 6, pp. 404-412.
- Karjalainen, L.P., Misra, R.D.K., Nayak, S., Venkatasurya, P.K.C., Ramuni, V., and Somani, M.C. (2010), 'Nanograined/Ultrafine-Grained Structure and Tensile Deformation Behavior of Shear Phase Reversion-Induced 301 Austenitic Stainless Steel', *Metallurgical and Materials Transactions A*, Vol. 41A, pp. 2162-2174.
- Kawasaki, M. (2010), 'Processing of Ultrafine-Grained Materials through the Application of Severe Plastic Deformation', *Metallurgical and Materials Transactions A*, pp. 1-11.
- Kolmogorov, A. N. (1937), *Izv. Akad. Nauk., USSR-Ser-Matemat*, Vol. 1, pp. 355-359.
- Kozeschnik, E., Pletenev, V., Zolotarevsky, N. and Buchmayr, B., (1999), 'Aluminium Nitride Precipitation and Texture Development in Batch-Annealed Bake-Hardening Steel', *Metallurgical Transactions A*, Vol. 30, pp.1663-73.
- Krishnan, K, Sahay, S.S., Singh, S. and Pal, D. (2006), 'Modelling the Accelerated Cyclic Annealing Kinetics', *Journal Of Applied Physics*, Vol. 100, pp 1-5.
- Krivobok, V.N. (1993), 'The book of Stainless Steels', *American Society for Steel Treating*.
- Kumar, B.R., Mahato, B., Sharma, S. and Sahu, J.K. (2009), 'Effect of Cyclic Thermal Process on Ultrafine Grain Formation in AISI 304L Austenitic Stainless Steel', *Metallurgical and Materials Transactions A*, Vol. 40A, pp. 3226-3234.
- Kumar, B.R., Das, S.K., Mahato, B. and Ghosh, R.N. (2009), 'Role of Strain-Induced Martensite on Microstructural Evolution during Annealing of Metastable Austenitic Stainless Steel', *Journal of Material Science*, Vol. 45, pp. 911–918.
- Kumar, B.R., Das, S.K., Sharma, S. and Sahu, J.K. (2010), 'Effect of Thermal Cycles on Heavily Cold Deformed AISI 304L Austenitic Stainless Steel', *Materials Science and Engineering A*, Vol. 527, pp. 875–882.
- Kumar, B.R., Sharma, S. and Mahato, B. (2010), 'Formation of Ultrafine Grained Microstructure in the Austenitic Stainless Steel and its Impact on Tensile Properties', *Materials Science & Engineering A*, pp. 1-22.

- Kumar, K.S., Swygenhoven, H. V. and Suresh, S. (2003), 'Mechanical Behavior of Nanocrystalline Metals and Alloys', *Acta Materialia*, Vol. 51, pp. 5743-5774.
- Lichtenfeld, J. A., Mataya, M. C. and Vantyne C. J. (2006), 'Effect of Strain Rate on Stress-Strain Behavior of Alloy 309 and 304L Austenitic Stainless Steel', *Metallurgical and Materials Transactions A*, VOL. 37, pp. 147-153.
- Low, J.R. and Garofalo, F. (1947), *Proc. Soc. Stress Analysis*, Vol. 4, pp. 16.
- Ludwigson, D.C. and Berger J.A. (1969), 'Plastic Behavior of Metastable Austenitic Stainless Steels', *Journal of The Iron and Steel Institute*, pp.63-69.
- Luo, H., Sietsma, J. and Van Der Zwaag, S. (2004), *Metallurgical and Materials Transactions A*, Vol. 35, pp. 1889–1898.
- Milititsky, M., Wispeleere, N. D., Petrov, R., Ramos, J.E., Reguly A. and Hänninen, H. (2008), 'Characterization of the Mechanical Properties of Low-Nickel Austenitic Stainless Steels', *Materials Science and Engineering A*, Vol. 498, pp. 289–295.
- Misra, R.D.K., Zhang, Z., Venkatasurya, P.K.C., Somani, M.C. and Karjalainen, L.P. (2010), 'The Effect of Nitrogen on the Formation of Phase Reversion-Induced Nanograined/ Ultrafine-Grained Structure and Mechanical Behaviour of a Cr-Ni-N Steel', *Materials Science and Engineering A*, pp. 1-26.
- Misra, R.D.K., Nayak, S., Mali, S.A., Shah, J.S., Somani, M.C. and Karjalainen, L.P. (2009), 'Microstructure and Deformation Behaviour of Phase-Reversion-Induced Nanograined/Ultrafine-Grained Austenitic Stainless Steel', *Metallurgical and Materials Transactions A*, Vol. 40A, pp. 2498-2509.
- Misra, R.D.K., Zhang, Z., Venkatasurya, P.K.C., Somani, M.C. and Karjalainen, L.P. (2010), 'Martensite Shear Phase Reversion-Induced Nano-Grained/ Ultrafine-Grained Fe–16Cr–10Ni Alloy: The Effect of Interstitial Alloying Elements and Degree of Austenite Stability on Phase Reversion', *Materials Science and Engineering A*, Vol. 527, pp.7779–7792.
- Muljono D., Ferry, M. and Dunne, D. P. (2001), 'Influence of Heating Rate on Anisothermal Recrystallization in Low and Ultralow Carbon Steels', *Material Science and Engineering A*, Vol. 303, pp. 90-99.
- Mythili, R., Saroja, S. and Vijayalakshmi, M. (2009), 'Study of Strain Induced Martensite formation in a Ti modified 316 Stainless Steel bellow by Transmission Electron Microscopy', *Transactions of The Indian Institute of Metals*, Vol. 62, Issue 6, pp. 573-579.
- Narutani, T. and Takamura, J. (1991), 'Grain-Size Strengthening in Terms of Dislocation Density Measured by Resistivity', *Acta Metallurgica et Materialia*, Vol. 39 pp. 2037-2049.
- Pickering F.B. (1976), *International Materials Reviews*, Vol. 21, pp. 2
- Radhakrishnan, B., Sarma, G.B. and Zacharia, T. (1998), 'Modelling the Kinetics and Microstructural Evolution during Static Recrystallization- Monte Carlo Simulation of Recrystallization', *Acta Materialia*, Vol. 46, No.12, pp. 4415-4433.
- Raghavan, V. (2004), '*Materials Science and Engineering*', PHI Learning Private Limited, pp. 285-286.

- Rajasekhara, S., Ferreira, P.J., Karjalainen, L.P. and Kyrolinen, A. (2007), 'Hall–Petch Behaviour in Ultra-Fine-Grained AISI 301LN Stainless Steel', *Metallurgical and Materials Transactions A*, Vol. 38A, pp. 1202-1210.
- Rajasekhara, S., Karjalainen, L.P., Kyröläinen, A. and Ferreira, P.J. (2009), 'Microstructure Evolution in Nano/Submicron Grained AISI 301LN Stainless Steel', *Materials Science and Engineering A*, Vol. 527, pp. 1986–1996.
- Rajasekhara, S. and Ferreira, P.J. (2011), 'Martensite to Austenite Phase Transformation Kinetics in an Ultrafine-Grained Metastable Austenitic Stainless Steel', *Acta Materialia*, Vol. 59, pp. 738–748.
- Roucoules, C., Pietrzyk, M. and Hodgson, P.D. (2003), 'Analysis of Work Hardening and Recrystallization during the Hot Working of Steel Using a Statistically Based Internal Variable Model', *Materials Science and Engineering A*, Vol. 339, pp.1-9.
- Sahay, S.S., Malhotra, C.P. and Kolkhede, A.M. (2003), 'Accelerated Grain Growth Behavior during Cyclic Annealing', *Acta Materialia*, Vol. 51, pp.339-346.
- Salishchev, G. A., Zaripova, R.G. and Zakirova, A. A. (2006), 'Structure and Properties of Stainless Steels Subjected to Severe Plastic Deformation', *Metal Science and Heat Treatment*, Vol. 48, Nos. 1 – 2, pp. 70-75.
- Samuel, E. I, Choudhary, B.K. and Bhanu Sankara Rao, K (2002), 'Influence of Temperature and Strain Rate on Tensile Work Hardening Behaviour of Type 316 LN Austenitic Stainless Steel' *Scripta Materialia*, Vol. 46, pp. 507–512.
- Samuel, K.G. and Rodriguez, P. (2005), 'On Power-Law Type Relationships and the Ludwigson Explanation for the Stress-Strain Behaviour of AISI 316 Stainless Steel', *Journal of Materials Science*, Vol. 40, pp. 5727–5731.
- Samuel, E. I., Choudhary, B.K. (2010), 'Universal Scaling of Work Hardening Parameters In Type 316l(N) Stainless Steel', *Materials Science and Engineering A*, Vol. 527, pp. 7457-7460.
- Shintani, T. and Murata, Y. (2011), 'Evaluation of the Dislocation Density and Dislocation Character in Cold Rolled Type 304 Steel determined by Profile Analysis of X-Ray Diffraction', *Acta Materialia*, Vol. 59, pp. 4314–4322.
- Somani, M.C., Juntunen, P., Karjalainen, L.P., Misra, R.D.K. and Kyrolainen A. (2009), 'Enhanced Mechanical Properties through Reversion in Metastable Austenitic Stainless Steels', *Metallurgical and Materials Transactions A*, Vol. 40A, pp. 729-744.
- Tadashi, M. (1997), 'Stainless steel: Progress in Thermomechanical Treatment', *Current Opinion in Solid State & Materials Science*, Vol. 2, pp.290-295.
- Takaki, S. and Tokunaga, Y. (1993), 'Ultra Grain Refining in Metastable Austenitic Stainless Steels', *International Conference on innovation in Stainless Steel*, Vol. 2, pp. 327-332.
- Valiev, R.Z., Estrin, Y., Horita, Z., Langdon, T.G., Zehetbauer, M.J. and Zhu, Y.T. (2006), 'Producing Bulk Ultrafine-Grained Materials by Severe Plastic Deformation', *Journal of Materials*, pp. 33-39.

- Yamamoto, S., Yokoyama, H., Abe, T. and Kobayashi, Y. (1993), 'Strengthening of Austenitic Stainless Steels by Thermo-Mechanical Control Process', *Material Processing and Research Centre*, Vol. 79, No. 4, pp. 524-530.