

**RECRYSTALLIZATION STUDIES
OF A LOW ALLOY STEEL IN THE ($\alpha + \gamma$) PHASE REGION**

BY

**ALANEME, KANAYO KENNETH
MME/99/3530**



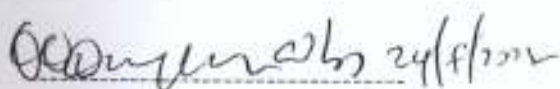
**A THESIS SUBMITTED TO
THE SCHOOL OF ENGINEERING AND ENGINEERING
TECHNOLOGY, FEDERAL UNIVERSITY OF
TECHNOLOGY, AKURE**

**IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR
THE AWARD OF MASTER OF ENGINEERING (M.ENG) IN
METALLURGICAL AND MATERIALS ENGINEERING
OF THE
FEDERAL UNIVERSITY OF TECHNOLOGY,
AKURE, ONDO STATE, NIGERIA**

AUGUST, 2002

CERTIFICATION

I certify that this work was carried out by, Alaneme Kanayo Kenneth, in the Department of Metallurgical and Materials Engineering, Federal University of Technology, Akure; and that, the work has not been submitted elsewhere for the award of a degree.



Engr. Prof. O.O. Onyemaobi
Head of Department



Prof. C. M. Kamma

(Major Supervisor)

Engr. Dr. John Ade Ajayi
(Co- Supervisor)



DEDICATION

This work is dedicated to God Almighty, the Being of beings; and to my father, Dr.S.O.Alaneme.



ACKNOWLEDGEMENT

It is with a deep heart of appreciation that I wish to acknowledge the immense contribution of my Major supervisor, Prof. C.M.Kamma, whose intellectual, and personal advice helped me in shaping my thought to embark on and fruitfully complete this work.

I am also grateful to my Co- supervisor, Dr John Ade Ajayi, for his patience to read through the work, and for his constructive criticism of my style of presentation.

I need also to say a big thank you to the Coordinator of the department, Engr B.O.Adewuyi, and all my lecturers and colleagues at the department, namely: Prof. O.O.Onyemaobi, Dr. A.Oni, Dr. J.O.Borode, Dr Oluwadare, Engr.J.A.Omotoyinbo, Dr.Adekale, Mr.A.Oyetunji, Mr.Oloruntoba, Mr.Aboginiye, Mr. Afolabi, Mr. Ogunniyi, Mr. Saliu, Mrs.Ola-Omole, Mrs. Lebile, Mrs. Ogundiminiyi, and our past Secretary, Mrs. Popoola; for their encouragement and assistance during the period I embarked on this work.

I do appreciate the effort of Mr. Alo and the other technical staffs of the Metallurgical and Materials Engineering department, Obafemi Awolowo University, Ile-Ife, for their assistance in the production of the photomicrographs used in this work.

I am particularly thankful to my friends: Kenneth Odoko, Thompson Okon, MC, and Henry Ovri, for the concern they showed while I was undertaking this research. Not forgetting also, Demola, Dewummi, and royal, who typed the



manuscript for the problem they encountered coping with the technical vagaries of this work.

My special thanks goes to my family for the patience and understanding they exercised during the course of my embarking on this work. I will not fail to thank my grand parents, Mr and Mrs A.D.Alaneme, for their parental guidance; also my uncles: Mr V.N.Alaneme, Mr C.Alaneme, and Mr A.A.Alaneme; my younger ones: Antoineth, Phylis, Victoria, Edith, Michael and the rest, for their brotherly love and affection. I also will not fail to mention the family of Mr Christain Alaneme, for their love and kindness.

Finally, I wish to acknowledge the several authors and researchers from whose works I have abstracted Information or quoted, and several other people who I cannot mention for sake of space, I owe you all an immense debt of gratitude.

Chapter

1.0 INTRODUCTION

2.0 LITERATURE REVIEW

2.1 Christianity

2.1.1 Transformation

2.1.1.1 Transformation

2.2 Mathematics

2.2.1 Homogeneity

2.2.2 Heterogeneity

2.3 Possible Cases

2.4 Intensity of

2.4.1 Relationship

TABLE OF CONTENTS

CONTENTS	PAGES
Title Page	i
Certification	ii
Dedication	iii
Acknowledgement	iv
Table of Contents	vi
List of Figures	ix
List of Plates	x
List of Appendix	xiii
Abstract	xiv
 Chapter	
1.0 INTRODUCTION	1
2.0 LITERATURE REVIEW	6
2.1 Classification of Steels	6
2.2 Transformation in Solids	8
2.2.1 Transformation Kinetics	8
2.3 Nucleation and Growth Mechanism	9
2.3.1 Homogenous Nucleation	10
2.3.2 Heterogenous Nucleation	12
2.4 Particle Coarsening	12
2.5 Instability Due to Strain Energy	14
2.5.1 Mechanism for the Storage of Energy	15

3.3.3	Samples Designation	42
3.3.4	Hardness Measurement	44
3.3.5	Metallography	44
3.3.6	Determination of Onset of Recrystallization / Austenite Precipitation	45
4.0	RESULTS AND DISCUSSION	46
4.1	Results	46
4.1.1	Rockwell Hardness (HRB) Vs Annealing Time	46
4.1.2	Starting Point Microstructure	46
4.1.3	Microscopic Investigation	46
4.1.4	Onset of Recrystallization	75
4.2	Discussion	75
4.2.1	Initial Microstructure	76
4.2.2	Interplay of Initial Microstructure, Deformation and Temperature on Recrystallization	77
4.2.3	Influence of Temperature on Recrystallization Kinetics	86
4.2.4	Influence of Deformation on Recrystallization Kinetics	87
5.0	CONCLUSION AND RECOMMENDATION	89
5.1	Conclusion	89
5.2	Recommendation	91
REFERENCES		92
APPENDIX		94

LIST OF FIGURES

- Fig. 2.1: Critical nucleus radius (Homogenous nucleation).
- Fig. 2.2: Effect of supercooling on critical radius
- Fig. 2.3: Orowan Looping of a dislocation past an array of precipitates of spacing λ .
- Fig. 2.4: Distribution of dislocation in a crystal deformed by bending.
- Fig. 2.5 Fe – Fe₃C phase diagramme.
- Fig. 2.6 Phases in Recrystallization Annealing Diagramme.
- Fig. 2.7 Technical application of recrystallization.
- Fig. 4.1 Hardness (HRB) Vs annealing time plot for normalised specimens at 740°C.
- Fig. 4.2 Hardness (HRB) Vs annealing time plot for normalised specimens at 760°C.
- Fig. 4.3 Hardness (HRB) Vs annealing time plot for tempered martensite specimens at 740°C.
- Fig. 4.4 Hardness (HRB) Vs annealing time plot for tempered martensite specimens at 760°C.

LIST OF PLATES

- Plate 1: Photomicrograph showing the normalised starting point microstructure.
(pearlite/ferrite) structure 860°C, 1 hour/ air.
- Plate 2: Photomicrograph showing the tempered martensite starting point microstructure; (M450)860°C, 1 hour/ H₂O +450°C, 1 hour/H₂O.
- Plate 3: Photomicrograph showing the as-deformed structure of P/20%/740°C.
- Plate 4: Photomicrograph showing the microstructure of P/20%/740°C, 15mins/H₂O.
- Plate 5: Photomicrograph showing the microstructure of P/20%/740°C, 1 hr/H₂O.
- Plate 6: Photomicrograph showing the as-deformed microstructure of P/50%/740°C.
- Plate 7: Photomicrograph showing the microstructure of P/50%/740°C, 1 min/H₂O.
- Plate 8: Photomicrograph showing the microstructure of P/50%/740°C, 15mins/H₂O.
- Plate 9: Photomicrograph showing the microstructure of P/50%/740°C, 30mins/H₂O.
- Plate 10: Photomicrograph showing the microstructure of P/50%/740°C, 1 Hr./H₂O.
- Plate 11: Photomicrograph showing the as-deformed microstructure of P/80%/740°C.
- Plate 12: Photomicrograph showing the microstructure of P/80%/740°C, 5mins/H₂O.
- Plate 13: Photomicrograph showing the microstructure of P/80%/740°C, 15mins/H₂O.
- Plate 14: Photomicrograph showing the microstructure of P/80%/740°C, 30mins/H₂O.
- Plate 15: Photomicrograph showing the as-deformed microstructure of P/20%/760°C.
- Plate 16: Photomicrograph showing the microstructure of P/20%/760°C, 5mins/H₂O.
- Plate 17: Photomicrograph showing the microstructure of P/20%/760°C, 15mins/H₂O.
- Plate 18: Photomicrograph showing the microstructure of P/20%/760°C, 30mins/H₂O.
- Plate 19: Photomicrograph showing the as-deformed microstructure of P/50%/760°C.
- Plate 20: Photomicrograph showing the microstructure of P/50%/760°C, 5mins/H₂O.
- Plate 21: Photomicrograph showing the microstructure of P/50%/760°C, 15mins/H₂O.
- Plate 22: Photomicrograph showing the microstructure of P/50%/760°C, 30mins/H₂O.

- Plate 23: Photomicrograph showing the microstructure of P/50%/760⁰C, 1 hr /H₂O.
- Plate 24: Photomicrograph showing the as-deformed microstructure of P/80%/760⁰C.
- Plate 25: Photomicrograph showing the microstructure of P/80%/760⁰C, 1 min/H₂O.
- Plate 26: Photomicrograph showing the microstructure of P/80%/760⁰C, 15 mins/H₂O.
- Plate 27: Photomicrograph showing the microstructure of P/80%/760⁰C, 30 mins/H₂O.
- Plate 28: Photomicrograph showing the microstructure of P/80%/760⁰C, 1 hr/H₂O.
- Plate 29: Photomicrograph showing the as-deformed microstructure of M450/20%/740⁰C.
- Plate 30: Photomicrograph showing the microstructure of M450/20%/740⁰C, 5 mins/H₂O.
- Plate 31: Photomicrograph showing the microstructure of M450/20%/740⁰C, 15 mins/H₂O.
- Plate 32: Photomicrograph showing the microstructure of M450/20%/740⁰C, 30 mins/H₂O.
- Plate 33: Photomicrograph showing the microstructure of M450/20%/740⁰C, 1hr/H₂O.
- Plate 34: Photomicrograph showing the as-deformed microstructure of M450/50%/740⁰C.
- Plate 35: Photomicrograph showing the microstructure of M450/50%/740⁰C, 5 mins/H₂O.
- Plate 36: Photomicrograph showing the microstructure of M450/50%/740⁰C, 15 mins/H₂O.
- Plate 37: Photomicrograph showing the microstructure of M450/50%/740⁰C, 30 mins/H₂O.
- Plate 38: Photomicrograph showing the microstructure of M450/50%/740⁰C, 1 hr/H₂O.
- Plate 39: Photomicrograph showing the as-deformed microstructure of M450/77%/740⁰C.
- Plate 40: Photomicrograph showing the microstructure of M450/77%/740⁰C, 1min/H₂O.
- Plate 41: Photomicrograph showing the microstructure of M450/77%/740⁰C, 5mins/H₂O.
- Plate 42: Photomicrograph showing the microstructure of M450/77%/740⁰C, 15mins/H₂O.

- Plate 43: Photomicrograph showing the microstructure of M450/77%/7400C, 1 hr/H₂O.
- Plate 44: Photomicrograph showing the as-deformed microstructure of M450/20%/760⁰C.
- Plate 45: Photomicrograph showing the microstructure of M450/20%/760⁰C, 5 mins/H₂O.
- Plate 46: Photomicrograph showing the microstructure of M450/20%/760⁰C, 15 mins/H₂O.
- Plate 47: Photomicrograph showing the microstructure of M450/20%/760⁰C, 1 hr/H₂O.
- Plate 48: Photomicrograph showing the as-deformed microstructure of M450/50%/760⁰C.
- Plate 49: Photomicrograph showing the microstructure of M450/50%/760⁰C, 1 min/H₂O.
- Plate 50: Photomicrograph showing the microstructure of M450/50%/760⁰C, 15 mins/H₂O.
- Plate 51: Photomicrograph showing the microstructure of M450/50%/760⁰C, 1hr/H₂O.
- Plate 52: Photomicrograph showing the as-deformed microstructure of M450/77%/760⁰C.
- Plate 53: Photomicrograph showing the microstructure of M450/77%/7600C, 1 min/H₂O.
- Plate 54: Photomicrograph showing the microstructure of M450/77%/760⁰C, 15mins/H₂O.
- Plate 55: Photomicrograph showing the microstructure of M450/77%/760⁰C, 30mins/H₂O.
- Plate 56: Photomicrograph showing the microstructure of M450/77%/760⁰C, 1hr/H₂O.

LIST OF APPENDIX

- Table 1: Rockwell hardness number for normalised starting point specimens with annealing time at 740°C .
- Table 2: Rockwell hardness numbers for normalised starting point specimens with annealing time at 760°C .
- Table 3: Rockwell hardness numbers for tempered martensite starting point specimens with annealing time at 740°C .
- Table 4: Rockwell hardness numbers for tempered martensite starting point specimens with annealing time at 760°C .
- Table 5: Quasi Kinetic Analysis Chart

ABSTRACT

An investigation of the technical viability of alloy development via thermomechanical processing, in steels, has been undertaken; by: a systematic study of the recrystallization kinetics in a low alloy steel in the ($\alpha+\gamma$) phase transformation region.

Two starting point microstructures (pearlite and tempered martensite) were prepared from as-received samples by the well known normalisation and tempering heat treatment processes. The initial microstructures were variously deformed by rolling and then subjected to recrystallization annealing at 740°C and 760°C . The soaking times were so chosen that the formation of the following microstructures was possible; recovered microstructures; partially recrystallized microstructures; and fully recrystallized microstructures. For each of the specimens, the start and progress of recrystallization and austenite precipitation were followed up by means of optical microscope as well as hardness measurement.

The results obtained show that varied microstructures can be produced and characterised, by manipulation of the test parameters (initial microstructure, degree of cold deformation, recrystallization annealing temperature, recrystallization annealing time), which were observed to influence the kinetics of the recrystallization/austenite precipitation process. In all, well defined duplex structures of recrystallized ferrite and martensite, having average grain sizes of $27.5\mu\text{m}$ and $10.6\mu\text{m}$, were obtained from the specimens having the pearlite/ferrite structure, deformed to a degree of eighty percent (80%), and subjected to annealing at 760°C for 30 minutes and 1 hour respectively, before quenching in water.



CHAPTER ONE

1.0 INTRODUCTION

Man's quest for materials that would perform specific tasks at optimum efficiency has over the years necessitated metallurgical and materials engineers to engage in discussions on materials selection; and explore the possibility of alloy development. From classical physical metallurgy, it has become common knowledge that the properties of metals and alloys are governed by their microstructures; and that these properties (mechanical, physical, chemical) can be changed by modification of the microstructural condition of the metals and alloys.

The modification of microstructure to effect property changes has over the years been done chiefly by alloying and heat treatment (Kamma, 1993). The former involves the use of alloying elements, which are added to improve upon specific properties, which are lacking in the metal; while the use of heat treatment involves mainly, the application of heat to bring about modification in the composition and distribution of phases in the metal/alloy (Rajan et al, 1988).

For quite some time, a lot of research has been centered on microstructure – property improvement through the use of heat treatment, because of the relative cost advantage it has over the use of alloying, which involves the use of alloying elements, which are often scarce and expensive. Also, alloying requires high processing technology, which for developing countries like Nigeria, is simply an unthinkable option for materials development, at least for now.

To maximize the advantage of heat treatment for alloy development, the use of "mechanical" means apart from the use of heat (thermal), has been incorporated into the heat treatment process (Ajayi, 1997). This involves the application of deformation process, either prior to heat treatment or in-situ; a process conventionally referred to as thermomechanical treatment.

The use of thermomechanical treatment has been greatly explored by development in the area of recrystallization; which in a loose sense, can be referred to as a process of "stress relaxation", as it involves the release of stored energy "stored" in a metal/alloy as an aftermath of plastic deformation. In a strict sense, it is a phenomenon, which controls the formation of structures, and determines the structure sensitive properties of metals and alloys, which are susceptible to the phenomenon (recrystallization). Thus the ever-growing interest in its study as the industries place increasing requirements on metals/alloys property performance.

The advancement of alloy development by thermomechanical treatment, has been greatly controlled by the progress in the study of recrystallization mechanisms; which Haasen (1997) has simply summarized as the most complicated, least understood, but technically important phenomena in metallurgy. However pessimistic this may sound, it is nevertheless obvious from the much available works on recrystallization mechanisms, that recrystallization thought could be said to have undergone a "Copernican Revolution": It was initially, believed that recrystallization is a metal softening process, and thus can only be applied in cases where softening of materials is required. Researches into recrystallization mechanism by Kamma and Hornbogen (1984), Humphreys and Hatherly (1995), and Hansen (2001) among many others, show that, except for pure

metals, other precipitation and ageing properties of metals/alloys could lead to a change in the recrystallization kinetics and thus might not necessarily lead to softening; but instead, could cause hardening.

This served as a point of departure for researchers like Hornbogen and Korster (1978), Kamma (1990), and Torres et al (2002) who working with different metals and alloys evolved a method for microstructure development, by studying the mechanisms of structural changes and ways of controlling them, making use of the principles of recrystallization and precipitation processes common to the different metals (alloys) they worked on.

The result of such study, has been applied by Kamma (1993), who noting the technological importance of steel to the Nigerian industries, proposed a methodology for design of microstructures in mild steel by a synthesis of the knowledge of recrystallization and the $(\alpha + \gamma)$ phase transformation; which would lead to the production of the duplex-structure steels of versatile application. Steels usually processed within the $(\alpha + \gamma)$ phase transformation region are referred to as dual phase steels. They have specifically been used in the production of automobile body parts and for truck manufacturing because of their good cold formability and strength.

These dual phase steels unlike the proposed duplex-structure steels are usually of very low carbon content (often $< 0.2\text{C}$ in wt%) as what is mostly desired is not more than 30% martensite on quenching from the $(\alpha + \gamma)$ region.

Unfortunately, little research efforts has been centered on using the advantage of recrystallization annealing in the $(\alpha + \gamma)$ region in "designing" microstructures for steels of higher carbon contents, which would lead to enhanced technological application of

steels for high ductility and deep drawing applications while at the same time maintaining a reasonably high carbon content. As technologically lucrative as this venture seems, much cannot be achieved if a phenomenological account of the mechanism through which these desired microstructural changes occur in the material. Xioyan et al (2001), have rightly acknowledged the exigency of such a task; they wrote that, "the explanation to the mechanisms of the recrystallization kinetics and the resulting microstructures as still not complete, owing to the intrinsic complexity of the different governing mechanism and their interactions". Thus, a thorough understanding of the kinetics and mechanisms through which these changes occur becomes imperative, as it would not only enable one to establish optimum conditions for the production of desirable microstructures, but would also enable one to a-priori, predict the properties of the produced microstructures and hence their applicability. Thus, the need to embark on a work of this nature, which aims at studying the kinetics of microstructural changes as a "means" (though it is an end) to the production of the duplex steels.

The successful completion of the research would enhance the use of "high" carbon steels for deep drawing and high ductility applications. Also, it will provide a theoretical framework for studying recrystallization mechanisms in multi-phase systems such as the ($\alpha + \gamma$) phase, which will serve as a necessary prolegomenon to future efforts on the production of duplex structures, especially in steels.

In the present work, a commercial low alloy carbon steel will be used as test material; considering the importance of low alloy steels in structural engineering applications, which are mostly used in the normalised, quenched, or tempered condition. The following starting point microstructures will be considered:

- (1) Normalised structure (pearlite/ferrite).
- (2) Tempered martensite structure consisting of fine dispersed cementite particles, capable of further growth during the subsequent recrystallization annealing treatment.

1.1 OBJECTIVES OF THE RESEARCH

The general objective of the research is to investigate the technical viability of alloy development via thermomechanical processing by recrystallization annealing in the ($\alpha+\gamma$) Phase region.

Other specific objectives are to:

- (a.) Investigate the influence of initial microstructure, degree of deformation and recrystallization annealing temperature on the kinetics of recrystallization.
- (b.) Investigate the influence of the superimposition of phase transformations on recrystallization kinetics.
- (c.) Determine optimum conditions for the production of duplex steels capable of enhancing ductility and deep drawing properties and at the same time maintaining a reasonably high carbon content in the low alloy steel.

Optical metallography and hardness measurements will be used as criteria for assessment of results obtained.

LITERATURE REVIEW

The study of steels is important because steels represent by far the most widely used metallic material primarily due to the fact that they can be manufactured relatively cheaply in large quantities to very precise specifications. They also provide an extensive range of mechanical properties from moderate ductility and toughness, to very high strengths with adequate ductility. Therefore, it is not surprising that iron and steels comprise well over 80% by weight of the alloys in general industrial use (Honeycombe, 1987).

2.1 Classification of Steels

Steels can be classified into two broad groups, namely plain carbon steels and alloy steels. Plain carbon steels are steel in which carbon is the main element which governs its properties, while alloy steels are those steels in which one or more elements are added to impart some peculiar characteristics. In alloy steels, carbon and alloying element, contribute towards properties of steels (Rajan et al, 1988). In the case of plain carbon steels, they can be classified on the basis of carbon content, structure and application. But for practical purposes, plain carbon steels are usually classified into low carbon steels ($0.2\%C - 0.3\%C$), medium carbon steels ($0.3\%C - 0.6\%C$), and high carbon steels ($0.6\%C - 1.7\%C$).

For alloy steels, they are considered when it is desired to improve upon the properties of plain carbon steels. The common deficiencies of plain carbon steels

which make the use of alloy steels inevitable include the following: low Hardenability; major loss of hardness on tempering; low corrosion resistance and oxidation resistance; and low strength at elevated temperatures. Alloy steels are classed into three groups:

i. Classification based on the quantity of alloying elements:

Under this classification, we have three groups

- (a.) Low alloy steels: - In which the total alloying content is less than about 5%.
- (b.) Medium alloy steels: - In which the total alloying content is in the range of 5 % – 10%.
- (c.) High alloy steels: - in which the total alloying content is above 10%.

ii. Classification by composition: - depending on the alloying elements, alloy steels are classified into nickel steels, chromium steels, Cr – Ni steels, and Cr – Ni – Mo steels.

iii. Classification by structure: various structures such as martensite, austenite, or bainite constitute steels. Accordingly, they are called martensitic steel, austenitic steel, bainitic steel.

iv. Classification by application: this includes corrosion resistant steel, heat resistant steel, magnetic steel, tool steel, electrical steel.

2.2 Transformations in Solids

Transformation in a solid involves the rearrangement of the atoms in the solid as a result of thermal or mechanical changes. This change in atomic arrangement often leads to changes in the original phases or microstructure; this subsequently, leads to changes in the physical and mechanical properties of the solid. Transformation in solids is affected by such factors as temperature, time and initial microstructure (Kamma and Hornbogen, 1984). Many metallurgical phenomena like decomposition, solidification, precipitation, and recrystallization are based on transformation.

2.2.1 Transformation Kinetics

As is well known, thermodynamic data or processes only provide us with information concerning the feasibility of a transformation to occur at prevailing conditions, or the driving force required for a transformation to occur. Metallurgical research interest is however, not only centred on the feasibility of processes occurring but also on the rate at which these processes proceed to completion. The study of how fast processes occur belongs to the science of kinetics. The rates of processes (transformation inclusive) are normally calculated with the help of the Arrhenius equation which is in accordance with the kinetics theory. The equation specifies that the probability of an atom reaching the activated state, that is, the energy barrier that has to be overcome by the atom before it can attain a lower energy level is given by $\exp(-\Delta G_a/KT)$, where K is known as the Boltzmann's constant, and ΔG_a is known as the activation energy barrier, (Porter

and Easterling, 1993). From the equation, it is seen that the rate at which a transformation occurs will depend on the frequency with which atoms reach the activated state, which is greatly enhanced by increasing temperature.

2.3 Nucleation and Growth Mechanism

From thermodynamics it is well known that for a reaction to occur the overall (net) energy change must be negative. Hence in a reaction in which the free energy of the parent phase (old phase) becomes higher than that of the product phase (new phase) through a change in temperature or pressure, a driving force for a phase change arises. In such an instance, the difference in bulk free energy between the product phase, and the present phase is negative, that is, $\Delta G < 0$. This condition is usually enhanced by thermal fluctuations which result in the formation of very tiny particles (containing only a few atoms) of the product phase within the parent volume. Such tiny particles have an interface that separates them from the parent matrix. They grow by the transfer of atoms across this interface. The transformation actually takes place in two steps; the first step involves the formation of tiny particles that are stable to further thermal fluctuations, and will not dissolve. This step is usually referred to as the nucleation step. The second step involves the increase in size of these tiny stable particles, and the process is referred to as growth process. Usually, the interface that separates the product and the parent phases has a surface energy. This energy has to be supplied during nucleation; it acts as an inhibiting factor for the phase change to occur. In general,

there are two types of nucleation; homogenous nucleation, and heterogeneous nucleation.

2.3.1 Homogenous Nucleation

Martin et al (1997), defined homogenous Nucleation as the nucleation that occurs without benefit of pre-existing heterogeneities. In this type of nucleation, the probability of nucleation remains constant throughout the volume of the parent phase. This requires greater super-cooling for stable nuclei to form; the greater the difference in bulk free energy between the product phase and parent phase, the greater the driving force to transform, that is, for a change of phase to occur.

If Δf is the energy change during nucleation of a spherical particle of radius r , we can have that

$$\Delta f = \frac{4}{3}\pi r^3 \Delta g + 4\pi r^2 \sigma \quad \dots\dots\dots (i).$$

where σ is the specific surface energy of the interface when a driving force exists for the phase change. For a spontaneous process, Δg is negative; but σ is always positive.

It is seen from Fig. 2.1 that below critical nucleus radius, r_c , particles of a new phase which form on the parent phase will tend to dissolve instead of grow. Cooling beyond the equilibrium temperature, that is, super-cooling, makes the value of Δg more negative, this leads to marked reduction in both the critical radius, and the energy barrier for nucleation as shown in Fig. 2.2. Thus there is a **greater probability that the necessary number of atoms to form a stable nucleus can attain the appropriate energy from some local energy fluctuations.** As a result, a

reaction which cannot nucleate at the equilibrium temperature occurs more readily when the degree of supercooling is great (Vanvlack, 1970).

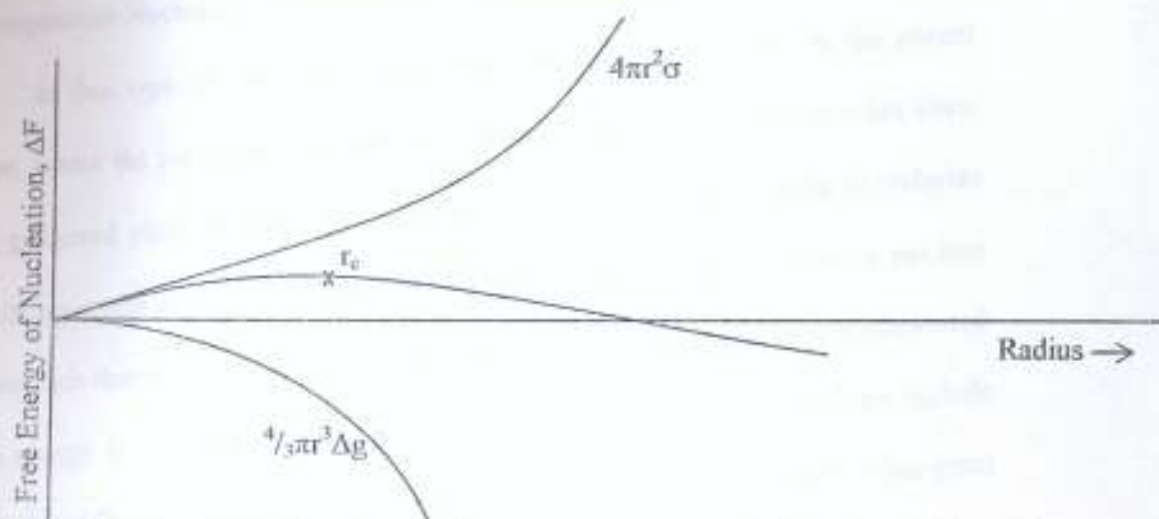


Fig 2.1: Critical nucleus radius (Homogenous nucleation) (vanvlack, 1970)

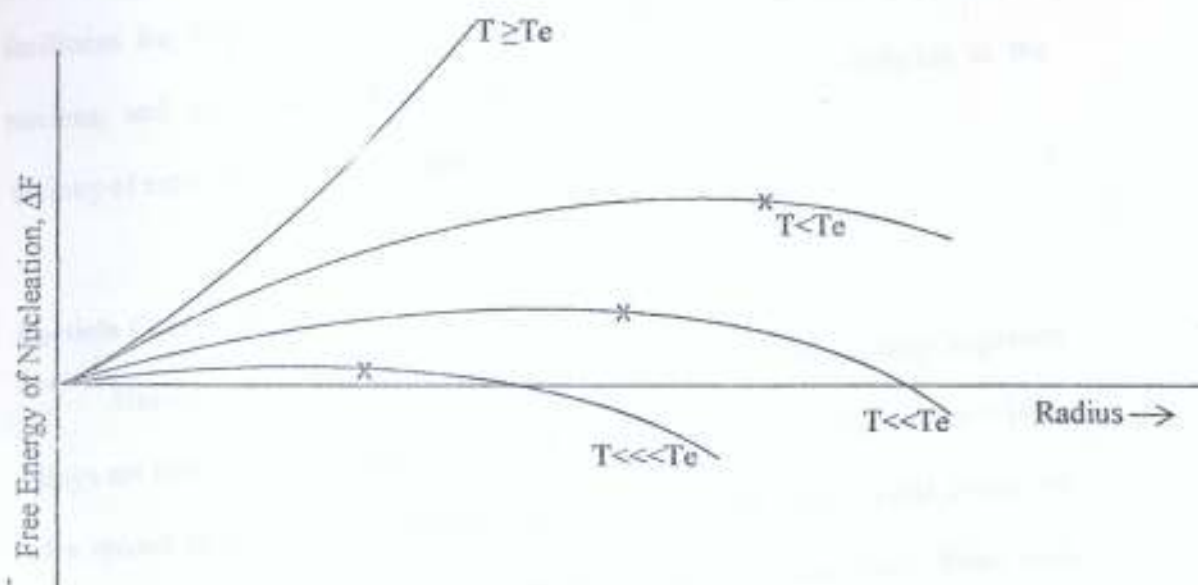


Fig 2.2: Effect of super-cooling on critical radius

(vanvlack, 1970)

2.3.2 Heterogeneous Nucleation

In this type of nucleation, there are certain preferred sites in the parent phase, where the probability of nucleation is much greater than that at other sites. The preferred sites are heterogeneities such as dislocations or grain boundaries which represent regions of higher than average free energy, so when a nucleus forms such that it replaces part of a boundary area or dislocation line the associated free energy is available to drive the reaction. Likely nucleation catalysts include grain boundaries, grain edges (three-grain intersection), grain corners (four-grain intersections), dislocations, stacking faults and the surfaces of impurity particles. The normal segregation of atoms to grain boundaries and dislocations also facilitates the formation of the appropriate group of atoms which constitutes a nucleus, and nucleation is further promoted by the enhanced diffusion in the vicinity of each type of defect (Martin et al, 1997).

2.4 Particle Coarsening

Haasen (1997), has rightly acknowledged that many technically important alloys are two-phase consisting of a matrix with a small volume fraction, $v_p > 10\%$, of a second phase. The microstructure of such two-phase alloys would always be unstable if the total interfacial free energy is not at a minimum. When such conditions prevail, there is a tendency for the small particles to dissolve and the resultant solute to precipitate on larger particles causing them to grow, thereby lowering the total interfacial energy (Smallman, 1992). However, such coarsening often produces an undesirable degradation of properties such as a loss of strength or

the disappearance of grain-boundary pinning effects (Porter and Easterling, 1993). As with grain growth, the rate of coarsening increases with temperature and is of particular concern in the design of materials for high temperature applications. The coarsening of precipitates which results as a result of high temperature treatment or longer ageing times makes the precipitate to become larger and thus stronger obstacles are formed. At this stage of the treatment, the interparticle spacing of the precipitates is such that dislocations are able to by pass them by looping between them. (Fig. 2.3).

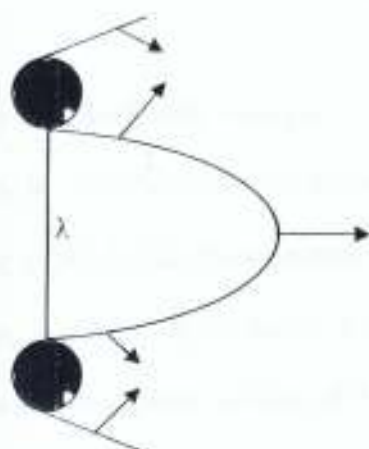


Fig. 2.3: Orowan Looping of a dislocation past an array of precipitates of spacing λ .

The increase in shear stress ($\Delta\tau$) over the value for the metal without precipitates, due to bowing of dislocations of line tension T_l between particles of mean planar separation, λ , is given, approximately, by the well-known Orowan equation,

$$\Delta\tau = 2T_l/\lambda b \quad \dots\dots\dots (ii)$$

The significance of this relationship is that any increase in the interparticle spacing, λ , will reduce the strength of the heat-treated alloy. Such a fall in strength with longer times of ageing, known as "overageing" is characteristic of precipitation hardening at least for artificial ageing. The most important mechanisms which cause over-ageing are: (i) the formation of more stable precipitates, since the more stable precipitates are usually more widely spaced; and (ii) the coarsening of the precipitate due to the interfacial free energy between the precipitate and the matrix usually referred to as "Ostwald ripening" (Martin et al, 1997).

2.5 Instability Due To Strain Energy

When a metal is subjected to plastic deformation by cold work, most of the work-done in the deformation process is converted into heat, but the remainder of this energy is stored in the metal. Cold-worked metals are unstable and, under favourable circumstances, undergo the processes of recovery and recrystallization, and during these processes stored energy is released. Normally, the free energy change associated with cold-work can only be evaluated through the first law of thermodynamics, given as:

$$\Delta U = Q - W \quad \dots\dots\dots (iii)$$

Where ΔU is the change in internal energy, Q is the heat associated with the deformation (positive if absorbed) and w is the work (positive if done on the body).



2.5.1 Mechanisms for the Storage of Energy

The interpretation of the mechanism of storage of energy arising from cold work is based on two concepts, namely the presence of lattice strains and of imperfections. However, x ray and calorimetric determination of the strain energy contribution to the total stored energy of deformed metals and alloys show that the energy contribution due to lattice strains are usually very small in comparison to the total stored energy (Martin et al, 1997); thus implying that the major part of total stored energy is due to the presence of lattice imperfections. These lattice imperfections are:

Point defects: When a crystal is deformed its resistivity (π_d) generally increases. The part of this extra resistivity that anneals out at temperatures below which appreciable changes in mechanical properties take place is believed to result from the resistivity due to point defects (π_p), and the other part to the resistivity of dislocations (π_d), that is,

$$\pi_d = \pi_p + \pi_d \quad \dots\dots\dots (iv)$$

Experiments of this type have shown that vacancies are produced by coldwork and that the concentration C depends on the amount of plastic strain ϵ according to the approximate relation,

$$C = 10^{-4}\epsilon \quad \dots\dots\dots (v)$$

These vacancies are presumed to form jogs produced on dislocation lines by intersecting dislocations, since it is observed that when the deformation is restricted to single slip the vacancy concentration (as shown by the change in electrical resistivity or density) is relatively small, while during double slip the resistance

increases and the density decreases. It should be pointed out that no single point defect (that is, vacancy or interstitial atom) has been positively identified as responsible for any particular part of the annealing spectrum of cold-worked metal. Detailed interpretation of annealing experiments is made very difficult due to the complexities of impurity effects, dislocation effects and point defect interactions. Vacancies must, however, be considered responsible for at least some energy. In cold-worked metals this energy is likely to be only a small fraction of the total stored energy.

- (ii) Linear defects (Dislocations): - The dislocation energy per unit length, E , can be obtained from the equation.

$$E = (\mu b^2 / 4\pi k) \ln(r/r_0) + \text{Core energy} \quad \dots\dots\dots (vi).$$

where μ is the shear modulus, r is the outer cut-off radius (which could be the specimen dimension), r_0 is the inner cut-off radius, b is the magnitude of the Burgers vector, and k is a constant (equal to unity for a screw dislocation and equal to $1 - \nu$, where ν is Poisson's ratio, for an edge dislocation). The core energy has been estimated as about equal to $\mu b^2 / 10$. Thus for a dislocation density, ρ , and neglecting the core energy, the elastic stored energy per unit volume, E_D , is then:

$$E_D = \rho (\mu b^2 / 4\pi k) \ln(r/r_0) \quad \dots\dots\dots (vii)$$

It follows that a uniform distribution of dislocations is unstable since the value of r , and hence the stored energy, can be reduced by clustering into stress screening arrays. As dislocations become arranged in dense tangles, various stress components can cancel, which leads to an effective lowering of the strain energy. Any attempt to estimate the value of E_D thus requires information on their density

and distribution. In crystals deformed within stage I of the stress-strain curve, the dislocation structure is mainly composed of isolated bundles or braids of long, relatively straight, primary edge dislocations lying on closely neighbouring primary glide planes. Stage II is marked by increasing numbers of secondary dislocations. Because most of the dislocations are confined to (iii) planes, at the higher dislocation densities occurring in stage II the structure on the slip planes become very complicated. In crystals deformed into stage III of the curve, the dislocation networks become even denser and the lattice tilts produced across them more pronounced. This dislocation distribution resembles that in stage II, especially for high deformations at low temperatures. The configuration at this stage can be described as dislocation sheets lying approximately parallel to the main glide, their separation decreasing with deformation. It is generally agreed that the dislocation distribution is highly non-uniform on the electron microscopical scale, the bundles or networks separating regions of the crystal almost free of dislocations. In polycrystals the dislocation structure produced by the deformation generally consists of irregular subgrains separated by tangled dislocation walls and with increasing deformation temperature, the cells become larger and the dislocation walls cleaner.

- (iii) Planar defects: - The structural change which occurs on increasing strain in fcc metals can be summarised as involving the following processes: the original dislocation "cell" structure first becomes subdivided by dense dislocation walls into regions taking up a large fraction of the grain. Then microbands of greater wall thickness evolve from the dense dislocation walls, and groups of microbands (called "transition bands")

removed, after which.

develop, and these have relatively high misorientations across them. At very high degrees of strain, such as in heavily rolled metals, "shear-bands" subdivide the structure; these are non-crystallographic, are not restricted to single grain, and sharp misorientations occurs across them. Large deformation twins, which are visible under the optical microscope, do not contribute appreciably to the stored energy, although twins on a fine scale and stacking faults are formed although twins on a fine scale, and stacking faults are formed during deformation, can account for a significant part of the stored energy, although twins on a fine scale, and stacking faults are formed during deformation, can account for a significant part of the stored energy.

2.5.2 Variables Affecting the Stored Energy

The reported values of the stored energy of cold work can be related to the energy expended during deformation (when this quantity is known), and a problem of special interest is the saturation of a metal with stored energy as the strain increases. The variables which affect the stored energy fall into two classes: those involving the deformation process (particularly the type and extent of deformation and the effect of temperature) and those involving the metal or alloy, such as its composition and metallurgical condition.

- (a) **Process Variables** - Many deformation processes are used in practice, including extension, compression, torsion, wire drawing and rolling, as well as cutting operations such as filing, drilling, and orthogonal cutting. Apart from differences in strain and strain-rate which may exist between them, more

fundamentally the simpler processes like extension involve negligible stress gradients, no frictional effects and (until necking begins) no redundant work. On the other hand, cutting and forming operations, such as wire drawing, and drilling, involve large stress and strain gradients, non-uniform strain rates, large frictional effects and appreciable amounts of redundant work. Research findings have shown that the simpler the mode of deformation, the lower the value of the stored energy. The effect of the temperature of working is a very important variable affecting the amount of stored energy. Work hardening rates usually increase with decreasing temperature, and it is also likely that, at lower temperatures, processes of energy release occurring during or immediately after the deformation are suppressed to a greater extent.

(b) **Material Variables:-** The identity of a metal is an important variable which has been found to influence the amount of stored energy. If several metals are deformed to the same extent using the same apparatus, it has been observed experimentally that the amount of stored energy decreases with decreasing melting points of the different test metals; though exceptions to this rule does exist (Martin et al, 1997). When foreign atoms are present in a metal, they allow larger amounts of energy to be stored during cold work as a result of the interactions between dislocations and particles of the foreign atoms (Smallman, 1992).

The grain size and orientation can also influence the stored energy at a given strain; for example, one would expect finer grain sizes to lead to higher values of stored energy because of the higher dislocation densities expected in fine grained material than in coarse grained material at the same strain due to the

"complexity effect". This requires multiple slip in the grains to allow changes in shape while maintaining continuity across grain boundaries; lattice deformation will thus be more complex in material with fine grains, because in a given grain, the close proximity of material in other orientations in adjacent deforming small grains. The presence of second phase particles has been found to raise the stored energy by an amount proportional to the volume fraction of particles and also to the magnitude of the applied strain. An understanding of these effects can be obtained from a consideration of the dislocation microstructures which develop in such systems during deformation. The stress strain curves of crystals containing a dispersed second phase may be classified according to whether the second phase deforms with the matrix or not. Work on systems containing a deformable second phase has been mainly confined to alloys containing a coherent precipitate, and it is found that the precipitate causes an increase in the yield stress due to coherency strains, and to several possible short range interactions, but it appears that the subsequent rate of work-hardening is not significantly different from that of the matrix alone, thus they are treated as a special form of solid solution. If the second phase particle is of the non-deforming type, a much higher dislocation density at a strain would be obtained in comparison with that of a single phase metal (Martin et al, 1997).

2.6 Influence of Heating on The Structure of Deformed Metals

Lattice defects are introduced into metals through deformations, irradiation, martensitic and diffusion controlled transformations; these processes generate dislocations, displacement cascades and grain boundaries respectively (Haasen, 1997). Whenever the kinetic conditions are favourable, the cold worked metal returns in the direction of the unworked or annealed state, the stored energy providing the driving force for the processes involved. When such a structural perfection occurs without the sweeping of the deformed structure by migrating high angle grain boundaries, the process is referred to as recovery. But if the crystal orientation or process of structural perfection in any region in the deformed material is effected by the passage of high angle grain boundaries through the material then it is referred to as recrystallization.

5.1 Recovery

This is a multi-stage process that includes all the elementary processes which take place prior to recrystallization. It is a process of structural perfection of a work hardened metal by redistribution and annihilation of point defects and by redistribution and annihilation of dislocations either without the formation of new boundaries or with the formation and migration of low angle boundaries only (Gorelik, 1981). The former is referred to as point defect annihilation, while the latter is referred to as polygonization.

(a.) Point – defect Annihilation: In this process, point defects are redistributed and their concentration is diminished due to their annihilation; and dislocations are distributed by conservative movement without forming new boundaries. Gorelik(1981), in his book reported that the activation energy required for this process is very low, and can occur at quite low temperatures, hundredths of the melting temperature (T_m). Point defect annihilation has been shown to reduce the resistivity of deformed metal on heating but has no influence on yield strength. The general mechanism for this process is believed to involve;

- i. The recombination of interstitial atom - vacancy pairs.
- ii. Migration of interstitial atoms, and
- iii. Migration of vacancies and bivalencies to traps.

(b.) Polygonization: In this process, dense dislocation networks formed by glide and interaction of dislocation as a result of coldwork, are rearranged and partially annihilated. The resultant effect is the lowering of the lattice strain energy as a result of the rearrangement of the dislocations into cell walls. The mechanism of polygonization could be illustrated by considering a single crystal lattice deformed by bending; this bending leads to the curvature of a region of the lattice as a result of the formation of excess edge dislocations parallel to the axis of bending as shown in fig2.4a below (Smallman, 1993). This leads to an increase in the energy of the crystal from the energy of dislocations proper and an additional energy of mutual repulsion of like dislocations located in common planes (Gorelik, 1981).

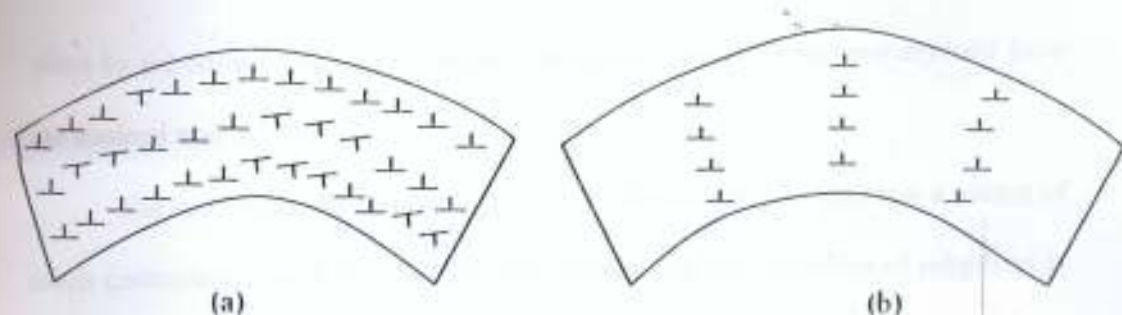


Fig 2.4: Distribution of dislocations in a crystal deformed by bending;

(a) before polygonization; (b) after polygonization

On heating, dislocations are regrouped and partially annihilated. Excess dislocations of one sign which are driven by elastic interaction, slip on the planes and then climb to form new (not existing before heating) tilt boundaries (cell walls) normal to the slip plane (Fig 2.4b) and sub boundaries formed in this way are typically of low curvature (low angle boundary). The regions of the crystal separated by these low angle boundaries are free of dislocations and represent subgrains (blocks or polygons).

During the recovery stage, the decrease in stored energy and electrical resistivity is accompanied by only a slight lowering of hardness.

2.6.2 Primary Recrystallization

At this stage of heating (annealing), the deformed lattice of cold worked metal is completely replaced by a new unstrained one of a higher structural perfection and lower energy by means of a nucleation and growth process. These practically stress-free grains grow from nuclei formed in the deformed matrix.

The orientation of the new grains differs considerably from that of the crystals they consume, so that growth process is regarded as incoherent, i.e. it takes

place by the advance of large – angle boundaries separating the new crystals from the strained matrix.

The mechanism of formation of recrystallization nuclei has been a source of much controversy; but it is now generally agreed that the formation of subgrains is a necessary preliminary stage to recrystallization. It is believed that recrystallization occurs in regions of high lattice distortions; this presupposes inhomogeneity in deformation which is a necessary precondition for recrystallization to occur. This deformation leads to the formation of excess dislocations of like sign which engenders polygonization of bent lattice regions in the lattice where the strain energy is lower than in the surrounding matrix (subgrains) (Smallman, 1993).

A large angle boundary, $\theta \approx 30^\circ - 40^\circ$, has a high mobility because the large lattice irregularities or “gaps” which exist in the boundary can easily transfer their allegiance from one crystal to the other. This subgrain is then able to grow at a much faster rate than the other subgrains which surround it and so acts as the nucleus of a recrystallized grain. Thus the recrystallization nucleus has its origin as a subgrain in the deformed microstructure. Whether it grows to become a strain – free grain depends on (i) The stored energy of cold work must be sufficiently high to provide the required driving force; the potential nucleus should have a size advantage over its neighbours; and it must be capable of continued growth by existing in a region of high lattice curvature, such as transition band, so that the growing nucleus can quickly achieve a high angle boundary (Smallman, 1993).

This stage sets in after primary recrystallization; and more strictly, as the growing recrystallization nuclei begin to collide with one another. Gorelik (1981), in his book, defined grain growth as the growth of some new grains at the expense of other (also new) grains under the conditions at which individual grains cannot grow preferably and exaggeratedly.

It is generally adopted that the principal thermodynamic driving force of grain growth is the trend to diminish the grain – boundary (surface) energy and counter balance the grain boundary (surface) tension of adjoining boundaries. Which is realized through shortening of the boundaries as they migrate until an equilibrium configuration of grain boundaries is established. The surface energy possessed by grain boundaries arises because its atoms have a higher free energy than those within the grains. Consequently, to reduce this energy a polycrystal will tend to minimize the area of its grain boundaries.

It has been established that boundaries move to the centres of their curvature and tend to move so as to have the angles at grain joints close to 120° . This occurs through preferable consumption of those grains which form sharper corners in triple joints, usually; boundaries migrate faster with an increased deviation of the angles from 120° .

In a single phase alloy, grain growth slows down sharply or practically ceases when most triple joints angles becomes equal to 120° and any grain has six ribs in its section, despite the fact that the absolute size of grains may be quite small. This is however applicable to substances possessing a weak anisotropy of

grain boundary energy that is f.c.c., b.c.c. and c.p.h substances. In the presence of disperse inclusions; grain growth can terminate even before the equilibrium angles have formed. It should be noted that as grains diminish in size, the rate of their consumption by neighbours grows. Thus, a stable structure is determined by angles in triple joints and by the number of sides in grain sections, rather than by the absolute size of the grains.

2.6.4 Secondary Recrystallization

Like grain growth, secondary recrystallization takes place when a material, in which primary recrystallization has occurred, is heated to a higher temperature, but differs from the former in that the grains grow not uniformly, but exaggeratedly, that is a portion of grains which have preferable conditions for growth increase in their size at the expense of the main portion of grains whose growth is impeded for some reason or other (Gorelik, 1981).

The process is similar to primary recrystallization in that the growth of secondary 'nuclei' also begins after a definite incubation period and that the size of the growing grains (before collisions begin) is linearly dependent on the time of heating. Secondary recrystallization inevitably involves equigranularity which can disappear only on completion of the process. In view of this, local inequigranularity can be regarded as a typical feature of unfinished secondary recrystallization. In the inequigranular structure, the largest grains (not numerous) are those which have formed in primary recrystallization; for some reason they have had the

advantage in growth over the main mass of grains and thus have become centres of secondary recrystallization. This can occur for the following reasons.

- (i) If some grains have a lower volume energy (lower concentration of defects) than other grains;
- (ii) If their size is larger than that of the remaining mass of grains;
- (iii) If their crystal orientation is different to that of the main mass of grains;
- (iv) If the crystal faces of these grains on the surface of a sheet or wire have a lower surface energy (at the metal – atmosphere boundary).

In the first case, the driving force of secondary recrystallization is a change in the free (volume, grainboundary, or surface) energy. At the end of primary recrystallization, grains may have different volume energies for a number of reasons. For instance, primary recrystallization nuclei formed through coalescence of subgrains may be less perfect than those formed through sub – boundary migration. Grains containing more disperse particles may have a higher dislocation density. If a material is subjected to light deformation or weak structural work – hardening, its various grains will usually be deformed unevenly. In heating, less strongly deformed grains will form at the expense of those which have been distorted more heavily, resulting in coarsening of the structure.

In the second case, difference in grain size and thus in grain boundary energy may be due to various causes. One of the probable causes is a non-uniform distribution and therefore a non-uniform dissolution of disperse particles. Owing to the latter fact, grain boundaries are freed and can migrate more quickly, so that the

grain boundary diminishes. Naturally, larger grains appear in less contaminated portions.

Another probable cause is a favourable crystal orientation of a growing grain relative to the surrounding grains of the textured matrix. In that case, the grain-boundary mobility becomes non-monotonic and dependent on the angle of boundary misorientation. It is then clear that, if a textured matrix has 'inclusions' of a certain number of differently oriented grains, the grains with most mobile boundaries will grow at the highest rate and will soon attain a size appreciably greater than that of the grains with a different orientation.

2.7 Design Criteria for Multiphase Microstructures

The processes of producing multiphase microstructures in mild steel can be classified under three separate mechanisms:

- (a) Phase – Transformation Annealing.
- (b) Recrystallization Annealing
- (c) Thermomechanical Treatment: involving mechanisms associated with phase transformation and recrystallization. Here, (a) will be devoted strictly to pure thermal application, while (b) and (c) will involve some superimposition of mechanical deformation to the thermal operation, at a pre-selected stage of the treatment.

2.7.1 Transformation in Steels

Iron is known to exist in various crystallographic configurations referred to as allotropic forms. These include the α -iron, γ -iron, δ -iron, with the crystal formations, bcc, fcc and bcc respectively. The well known Fe – Fe₃C constitution diagram for carbon steel (Fig. 2.5) shows the allotropic changes occurring in iron in the temperature range 0°C - 1536°C, as well as the temperature dependence of carbon solubility and allotropic forms of iron is effectively utilized in the production of conventional ferrous multiphase microstructures. In fact, the traditional heat treatment phenomena associated with Normalization Annealing, Tempering and Martensitic transformation have the Fe – Fe₃C phase diagram as their operational tool.

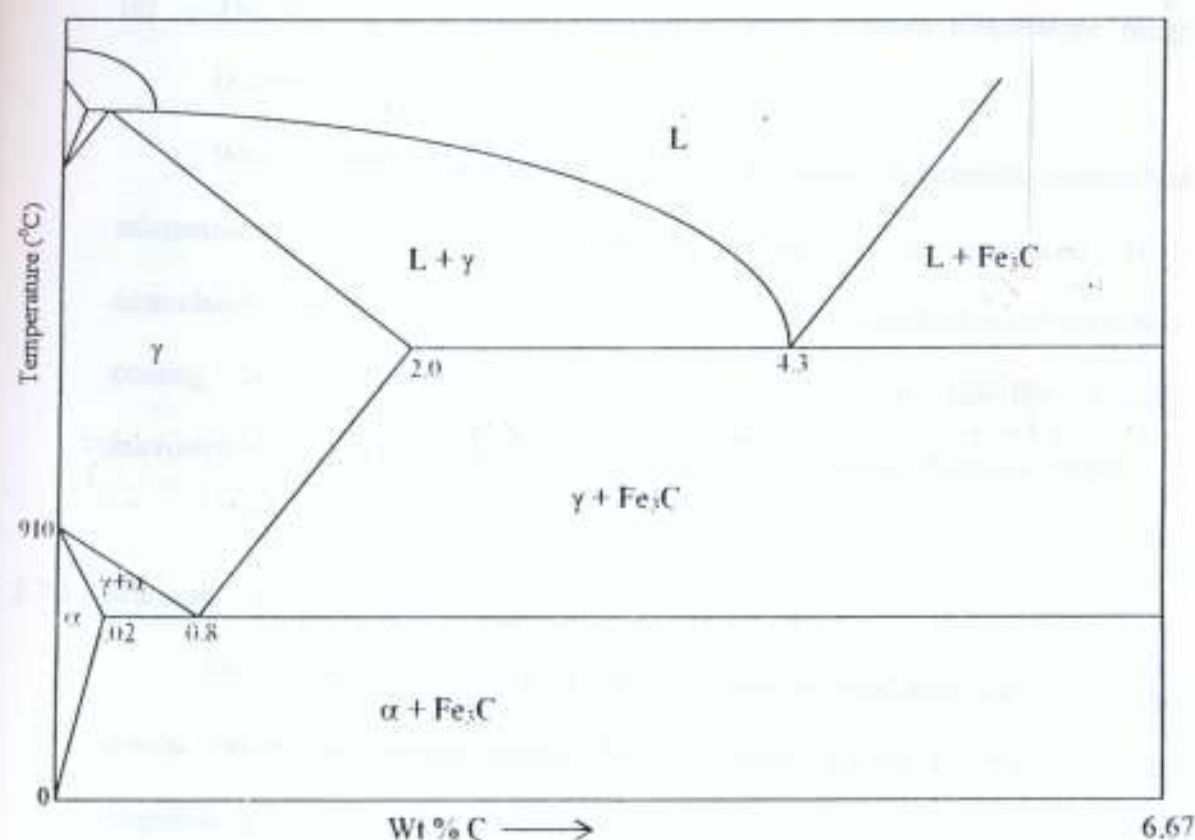


Fig. 2.5: Fe – Fe₃C Phase diagram

2.7.2 Normalization

The production of normalized microstructures in carbon steels utilizes the enhancement of mechanical and physical properties inherent in the two-way transformation of the α_{Fe} and γ_{Fe} allotropes.

Cooling: $\gamma_{Fe} \longrightarrow \alpha_{Fe}$

Heating: $\alpha_{Fe} \longrightarrow \gamma_{Fe}$

The quality of the normalized microstructure is influenced by:

- The initial microstructure of the α_{Fe} .
- The rate of heating from room temperature to the γ_{Fe} – temperature.
- The austenitization temperature.
- The time of holding at the austenitization temperature.
- The cooling rate within the austenitization – room temperature range (Kamma, 1993).

With the help of appropriate choice of the above parameters, normalized microstructures of desired inter-lamellar-spacing can be produced. High austenitization temperatures, long hold-on times during austenitization and very slow cooling favour coarser pearlitic microstructures. Fine lamellar pearlite microstructures have higher tensile qualities and machinability (Kamma, 1993).

2.7.3 Martensite Microstructure

The martensitic structure of ferrous alloys is associated with very high tensile values and brittle nature, brought about mainly by the following contributory factors:

- (a) Interstitial Carbon atoms
- (b) Dislocation reactions
- (c) Phase-transformation associated changes such as internal stresses.

Successful production of martensitic microstructures depends largely on:

- (i) Carbon content of the given carbon steel; it is difficult to obtain martensitic structures in carbon steels of very little carbon content as this would necessitate an extremely high cooling rate,
- (ii) extent of the carbon dissolution during the austenitization stage,
- (iii) quenching characteristics of the quenchant.

Depending on the appropriateness of application manipulation of the above parameters, one can come up with the following possible martensitic microstructures.

- (a) 100% martensitic structures (M): Ideal parameter selection.
- (b) M + intermediate phases: Inadequate quenching rate.
- (c) M + retained austenite: existence of alloying elements such as Chromium.
- (d) M + segregated carbon atoms: martensitic transformation atom with subsequent natural ageing.
- (e) M + pearlite structures (P): incomplete Austenitization before quenching.
- (f) M + P + Fe_3C : by hypereutectoid steels and insufficient austenitization time.



2.7.4 Tempered Microstructures

Tempering involves the subjection of a martensitic structure to some heating at temperature usually below the A_1 – point. This treatment has the dual effect of

- (a) Turning the crystallographic character of the martensite from the bct to a pearlite bcc.
- (b) Converting the interstitial carbon atoms of the martensite into a dispersion of Fe_3C in a pearlitic Fe-matrix. This gives rise to a partial relief of internal stress of microstructures and consequent improvement of ductility in the tempered structure. On the other hand, the emergent, dispersed Fe_3C – particles (which can be needle-like at low tempering temperatures) interact with dislocations associated with the martensitic structures giving rise to a cold-working related strengthening effect in the tempered microstructure.

The properties of a tempered microstructure can be effected by appropriate choice of the:

- (i) Starting point martensitic structure,
- (ii) tempering temperature.

A modification of the tempering treatment is the prolonged heating of the lamellar pearlitic structure around the A_1 point temperature range of the Fe- Fe_3C phase diagramme. This heat changes the lamellar structure to a globular one which is softer and more ductile.

2.8 Thermomechanical Treatment

The subjection of a form of deformation to a material undergoing or about to undergo a thermal treatment can have one of two possible effects.

- (a) The ensuing microstructure, while retaining its original crystallographic structure, is of a different texture. This situation is encountered in recrystallization mechanisms.
- (b) The original microstructure undergoes a phase transformation and the ensuing microstructure is changed crystallographically. This is an exercise which involves meaningful application of transformation diagrams (for example; phase diagrams, TTT diagram) and effective utilization of the interrelationship between degrees of deformation and reaction kinetics. This thermomechanical treatment could be used in the production of microduplex structures in carbon steels and can be seen as a potentially result-oriented tool in future alloy development efforts.

2.8.1 Recrystallization Annealing

The conventional notion of primary recrystallization involves a discontinuous reaction, leading to the production of required grains at temperatures above the recrystallization temperatures; a minimum amount of deformation is usually necessary to trigger off the reaction and the recrystallization duration is seen to decrease with increasing degree of deformation; particles appear to act as nucleation sites for recrystallization and are therefore deemed to accelerate recrystallization. The ensuing recrystallized grains are more ductile than the initial

microstructures. Consequently, recrystallization annealing appears to be synonymous with grain softening heat treatment. The hardness Vs recrystallization time is shown in Fig. 2.6.

However, current recrystallization studies by Kamma and Hornbogen (1984) Humphreys and Hatherly (1995), Hansen (2001), amongst others, has shown far-reaching modification of the earlier findings:

- (a) Second-phase particles can accelerate or retard recrystallization, depending on their size, morphology and distribution.
- (b) At very high deformations ($> 80\%$) recrystallization can be retarded, if the deformation/particle dispersion condition leads to a homogenization of the matrix.
- (c) Recrystallization can represent a grain-strengthening heat treatment if, for instance,
 - Recrystallization and grain-growth are inhibited by the stabilising action of the second-phase particles.
 - Recrystallization involves a continuous reaction (in-situ recrystallization).
 - The activation energy of primary recrystallization is appreciably reduced by prior recovery effects.

Accordingly, recrystallization annealing can be used to produce a wide range of microstructures (Fig. 2.7) by systematic and appropriate choice of relevant parameter, notably:



- (a) Second-phase particles of various dispersion,
- (b) degree of deformation,
- (c) recrystallization mechanism: continuous or discontinuous,
- (d) recrystallization annealing temperature.

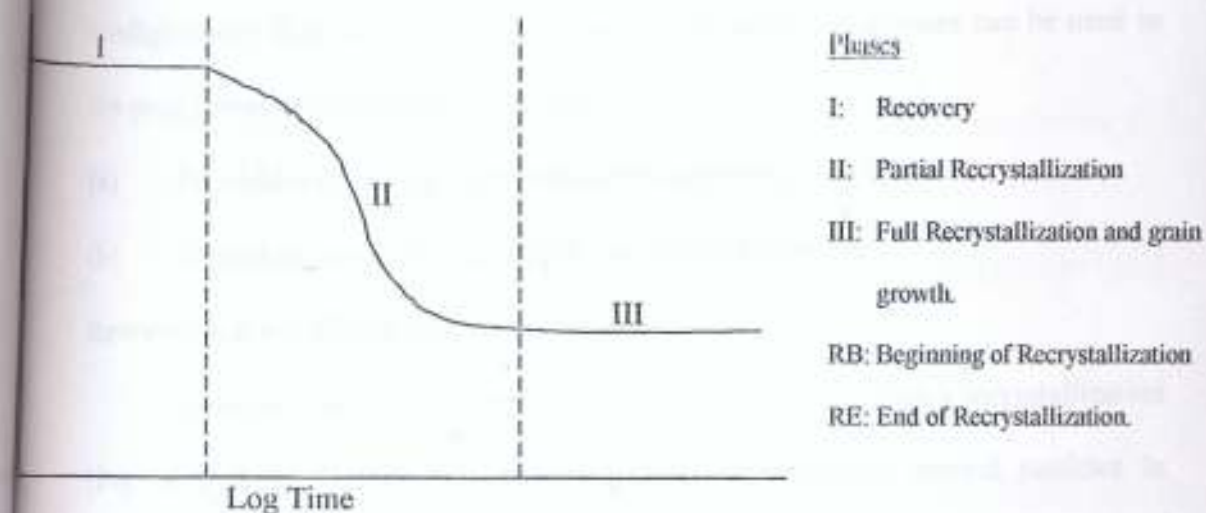


Fig. 2.6: Phases in Recrystallization Annealing Diagramme

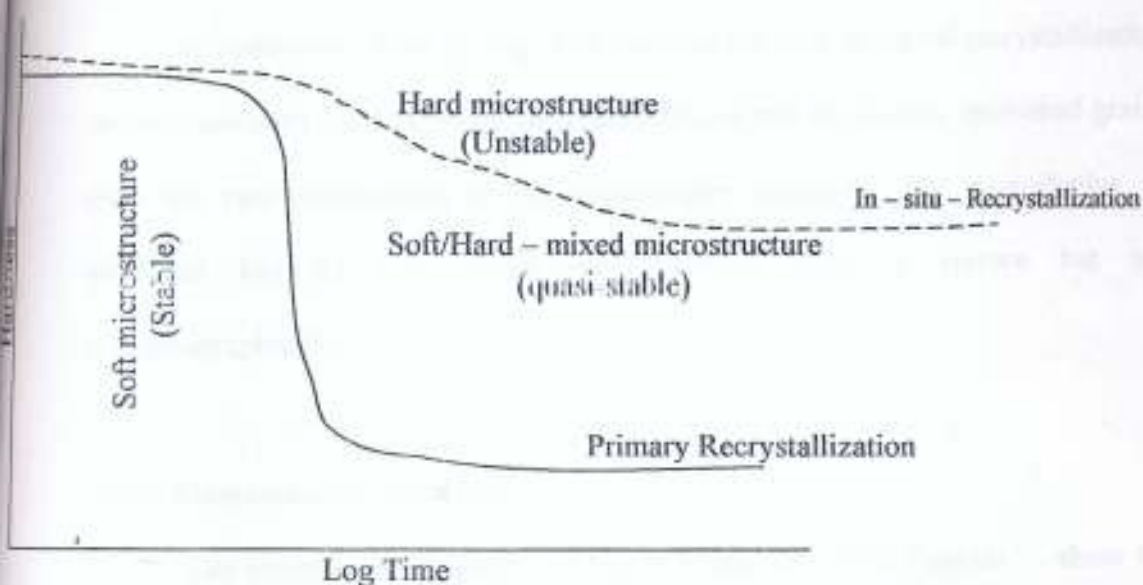


Fig. 2.7 Technical Application of Recrystallization

11.2 Microduplex-Structure Annealing

A microduplex structure can be seen as a 2-phase metallurgical structure of comparable volume-fraction which could differ either in texture or crystallographic configuration. Basically, two thermomechanical treatment avenues can be used in the production of microduplex structure:

- (a) Procedure involving recrystallization annealing.
- (b) Procedure involving sequential phase transformation.

Recrystallization Annealing Procedure

Here use can be made of the recovery-zone of the primary recrystallization (Fig. 2.6, Zone I). The relevant parameters (temperature, second particles in appropriate dispersion and degree of deformation) are so chosen that sharp, well-developed subgrains amidst deformed structures are produced.

Alternatively, Zone II (Fig. 2.6) showing the area of partial recrystallization can be used with success. Here, the recrystallized and the merely recovered grains from the two components of the microduplex structure. The microduplex so produced has its two components differing only in texture but not crystallographically.

Phase Transformation Procedure

The constitution diagram (Fig. 2.5) and the TTT diagram show the zones of existence of the various allotropic forms of iron.

γ_{Fe} -zone: Quenching from the γ -zone, one can get 100% martensite by complete austenitization, Austenite $\longrightarrow$ martensite (quenching). By incomplete

austenitization, therefore, one would expect a mixture of pearlite and martensite after quenching. A microduplex so produced would be made up of:

- (i) Untransformed pearlitic phase during the austenization stage,
- (ii) martensitic phase, arising from the quantity of α -phase transformed at the austenitization stage.

By careful choice of appropriate austenization duration, the correct proportion of pearlite/martensite can be obtained.

$\alpha+\gamma$ zone: By appropriate choice of the time of holding at the $\alpha+\gamma$ zone, an α/γ volume fraction can be obtained that compares favourably with the components of a microduplex structure. On quenching, the α -phase remains pearlitic; the γ -phase is converted to martensite. The microduplex structure therefore is made up of two different phases, namely pearlite and martensite.

Transformation and Recrystallization:

A combination of the recrystallization and α/γ -phase transformation processes in the $\alpha+\gamma$ zone of the Fe-Fe₃C diagramme will equally produce microduplex structures that have improved mechanical properties. Modifications of the microstructures can also be obtained by delaying the onset of the $\alpha \rightarrow \gamma$ transformation in the $\alpha+\gamma$ zone. This can be achieved by considerations of the kinetics of both $\alpha \rightarrow \gamma$ transformation and recrystallization reactions in the $\alpha+\gamma$ zone which Hornbogen and Koster (1978) have reviewed:

$$t_r = K_r \exp (Q_r/KT) \quad \dots\dots\dots \text{(viii)}$$

$$t_p = K_p \exp [(Q_n + Q_D)/KT] \quad \dots\dots\dots \text{(ix)}$$

t_r, t_p = times to beginning of recrystallization and transformation +
respectively.

K_r, K_p = recrystallization and transformation constants, containing the geometrical terms, entropy terms and driving forces for the reactions.

Q_r = Activation energy for the beginning of recrystallization.

Q_D = Activation energy for the diffusion of foreign atoms.

Q_n = Activation energy for the nucleation.

K = Boltzmann constant.

T = Absolute temperature.

From the above, it is seen that except recrystallization is complete before the precipitate is nucleated, precipitation and recrystallization exert a mutual influence upon each other (Hornbogen and Koster, 1978). Thus precipitate particles hinder the formation and migration of recrystallization fronts', while the lattice defects themselves promote the nucleation of precipitates. Consequently, by appropriate choice of degree of deformation and temperature, the precipitation reactions can be used to control the size of recrystallized grains and hence regulate the relative volume fraction of the duplex components, ferrite and austenite.

Furthermore, the extent of prior recovery in the austenitization stage can be used to influence the incubation time at the $(\alpha+\gamma)$ zone and hence, indirectly the kinetics of both the transformation and recrystallization reactions at the $(\alpha+\gamma)$ zones.

CHAPTER THREE

3.0 EXPERIMENTAL PROCEDURES

3.1 Equipment

The equipment used for this research are: Miniature Cold Rolling Machine, Muffle Furnace, Power Hacksaw Machine, Chemical Analyser, Grinding and Polishing Machines, Optical Microscope With Camera, Rockwell Hardness Tester, Milling Machine, and Computer with Printer.

3.2 Materials

3.2.1 Materials Procurement

The material for this investigation was a low alloy steel obtained from Delta Steel Complex Aladja in the form, of scrap billets.

3.2.2 Chemical Analysis

The chemical composition of the low alloy steel was determined at Universal Steels Ogba, Ikeja, Lagos; with the aid of a spark analyzer that uses emission spectography and the pin spark method for the analysis. The chemical composition of the low alloy steel in wt% is as follows; C(0.4); Si(0.26); Mn(1.19); P(0.0419); S(0.0024); Cr(0.13); Ni(0.035); Mo(0.0042); V(0.0017).

2.3 Normalising of Materials

All heat treatments were carried out at the Metallurgical and Materials Engineering laboratory of the Federal University of Technology, Akure. The first Heat treatment operation performed was the initial normalizing of the low alloy steel for the investigation, using a muffle furnace. This was done to soften the steel for subsequent machining operations. The steels were austenitised at 860°C for one hour, and then allowed to cool in air.

2.4 Machining Of Samples

The machining operation was performed in the General Engineering Workshop of the Engineering Faculty of University of Lagos, Akoka. The low alloy steel was cut into ten pieces of different thickness. The steel pieces of 2.5mm; 3.07mm; 4mm; 6.67mm; and 10mm were meant to undergo normalising treatment while another set of five pieces, 2.5mm; 3.07mm; 4mm; 6.67mm and 8.7mm were meant to undergo tempering treatment.

Methods

2.1 Production of Initial Microstructures

The machined specimens were initially normalized at 860°C for 1 hour and then cooled in air using a muffle furnace, so as to restore their original condition and to induce homogeneity in their structure. After normalizing treatment, each of the five sets of five machined samples (section 3.2.4) was used to prepare two

different microstructures serving as starting point phases. The treatments were performed as follows:

- (i) Tempered Martensite: The five test pieces for the tempering treatment (section 3.2.4) were soaked for 1 hour at 860°C and then quenched in water. The quenched test pieces were subsequently reheated at a tempering temperature of 450°C for 1 hour before quenching in water.
- (ii) Normalized (pearlitic/ferritic) microstructure: also the five test pieces for the normalising treatment (section 3.2.4) were soaked for 1 hour, but allowed to cool in air to produce a pearlite/ferrite equilibrium microstructure.

3.3.2 Thermomechanical Treatment

The cold rolling operation was performed at the General Engineering Workshop of the Faculty of Engineering, University of Lagos, Akoka, with the aid of a miniature cold rolling machine. The samples of different initial microstructures and different thicknesses were all deformed to a uniform thickness of 2mm. The samples of tempered martensite of initial thicknesses 2.5mm, 3.07mm, 4mm, 6.67mm and 8.7mm were deformed to 2mm implying that they were subjected to 20%, 33%, 50%, 70% and 77% deformation respectively; while the normalized test pieces of thickness 2.5mm, 3.07mm, 4mm, 6.67mm and 10mm were also deformed to 2mm; implying 20%, 35%, 50%, 70% and 80% deformation respectively.

After the cold deformation operation, the test pieces were subjected to recrystallization annealing at temperatures of 740°C and 760°C . The samples

annealed at 740°C were variously held for 1minute, 5minutes, 15minutes, 30minutes and 1hour before quenching in water; while the samples annealed at 760°C were held for 30secs, 1minute, 5minutes, 15minutes, 30minutes and 1hour respectively, before quenching in water.

3.3.3 Samples Designation

After the initial microstructure preparation, the ensuing starting point microstructures were designated as follows.

- (i) M450: 860°C , 1hr/ H_2O + 450°C , 1hr/ H_2O ; which means that the tempered initial microstructure was obtained by soaking the steel samples for one hour at 860°C then quenching in water; the steel sample were then subsequently reheated to 450°C and held for one hour before quenching in water.
- (ii) P: 860°C , 1hr/air; which means that the normalized initial microstructure was obtained by soaking the steel sample for 1 hour at 860°C , but allowed to cool in air.

For the samples subjected to thermomechanical treatment, the following notations were used to express the nature of the thermomechanical treatment carried out on the test specimen.

- (iii) M450/77%/ 740°C , 30mins/ H_2O ; means that the starting point specimen after 77% deformation, was subjected to recrystallization annealing at 740°C and held for 30minutes before quenching in water.

- (iv) P/20%/760°C, 1min/H₂O, would mean that the normalized starting point specimen, after 20% deformation, was subjected to recrystallization annealing at 760°C and held for 1 minute before quenching in water.
- (v) M450/50%/760°C, 15mins/H₂O, would mean that the tempered martensite starting point specimen, after 50% deformation was subjected to recrystallization annealing at 760°C and held for 15 minutes before quenching in water.
- (vi) P/35%/740°C, 5mins/H₂O; would mean that the normalized starting point specimen, after deformation was subjected to recrystallization annealing at 740°C and held for 5 minutes before quenching in water.

Finally, for samples of the same initial microstructure, subjected to the same types of thermomechanical treatment but held for different times, the following notation was used.

P/20%/740°C, 0-1hr, would mean the following microstructure and/or hardness Vs annealing time profile was obtained when the normalized starting point specimen, deformed by 20%, and subjected to recrystallization annealing at 740°C was held at different times from zero seconds to one hour.

M450/50%/760°C, 0-1hr, would mean that the following microstructure and/or hardness Vs annealing time profile was obtained when the tempered martensite starting point specimen, deformed by 50%, and subjected to recrystallization annealing at 760°C was held at different times from zero seconds (as-deformed state) to one hour.



3.3.4 Hardness Measurement

The hardness test was performed at the Stress Analysis Laboratory of the Mechanical Engineering Department of University of Lagos, Akoka using a Rockwell Hardness Tester. The hardness test was used to establish the different stages of recrystallization/phase transformation (austenite precipitation) by means of hardness Vs annealing time plots. The results of the hardness test are shown in tables 1 – 4 in appendix and the plots of the hardness Vs annealing time are shown in Fig. 4.1 – 4.4. The specimens for the hardness test were ground and polished to ensure accurate hardness readings. For convenience, the logarithm of time is used in the time axis (x axis; abscissa).

3.5 Metallography

The microstructural examination of specimens was performed at the Metallurgical and Materials Engineering laboratory of Obafemi Awolowo University, Ile-Ife. To ensure microstructural consistency, the same specimens used as hardness test pieces were reground and repolished for microstructural examination, using a grit grinder with emery papers (220, 320 and 500 grit) and a polishing machine (using water and aluminium paste) respectively. The polished specimens were then etched with a 2% Nital solution for 10 seconds and quickly rinsed with water. After these metallographic treatments, the beginning, progress and end of recrystallization, as well as other precipitation, and growth processes were monitored with the help of a light microscope. In-situ examination of the

structures were first carried out, before the respective photomicrographs were taken; also, the average grain size of well defined crystal structures was determined making use of the linear analysis method. The photomicrographs obtained are shown in plates 1 – 56.

3.3.6 Determination of Onset Of Recrystallization/Austenite Precipitation

With the aid of hardness Vs annealing time plots (Fig. 4.1 – 4.4) and the photomicrographs (Plates 3 – 56), The time lapse before the beginning (t_{pb}) and end (t_{pe}) of austenite precipitation as a function of degree of deformation and recrystallization annealing temperature was determined. For experimental convenience, the time lapse at which the first sign of recrystallization is observed on the photomicrograph is taken as the time for recrystallization initiation (t_{RI}), while the time lapse just prior to the beginning of the rise in the hardness value in the hardness Vs annealing time plot is taken as the time for the “beginning of precipitation”, and the time lapse after which a fall in hardness is experienced after the initial rise on the hardness Vs annealing time plot is taken as the “end of precipitation” of austenite. The results are shown in table 5 of appendix, and as a result of the obvious approximations, the table is referred to as Quasi-kinetic analysis chart.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.0

4.1 Results

4.1.1 Rockwell Hardness (HRB) Vs Annealing Time:

Tables 1 to 4 in appendix show the results of the variation of hardness obtained with annealing time; the results from the tables were used to plot the graphs shown in Fig. 4.1 to Fig. 4.4 on pages 47 - 50.

4.1.2 Starting Point Microstructure

Plates 1 and 2 show the photomicrographs of the two pretreatments used as starting-point microstructures in this work. Plate 1 consists of an equilibrium structure of lamellar pearlite (dark regions) with ferrite (white), while Plate 2 shows a fine dispersion of needle like Fe_3C particles (tempered martensite).

4.1.3 Microscopic Investigation

The photomicrographs of optical microscopic investigation of the kinetics of the recrystallization process for the different thermomechanical treatment are shown in Plates 3 to 56.

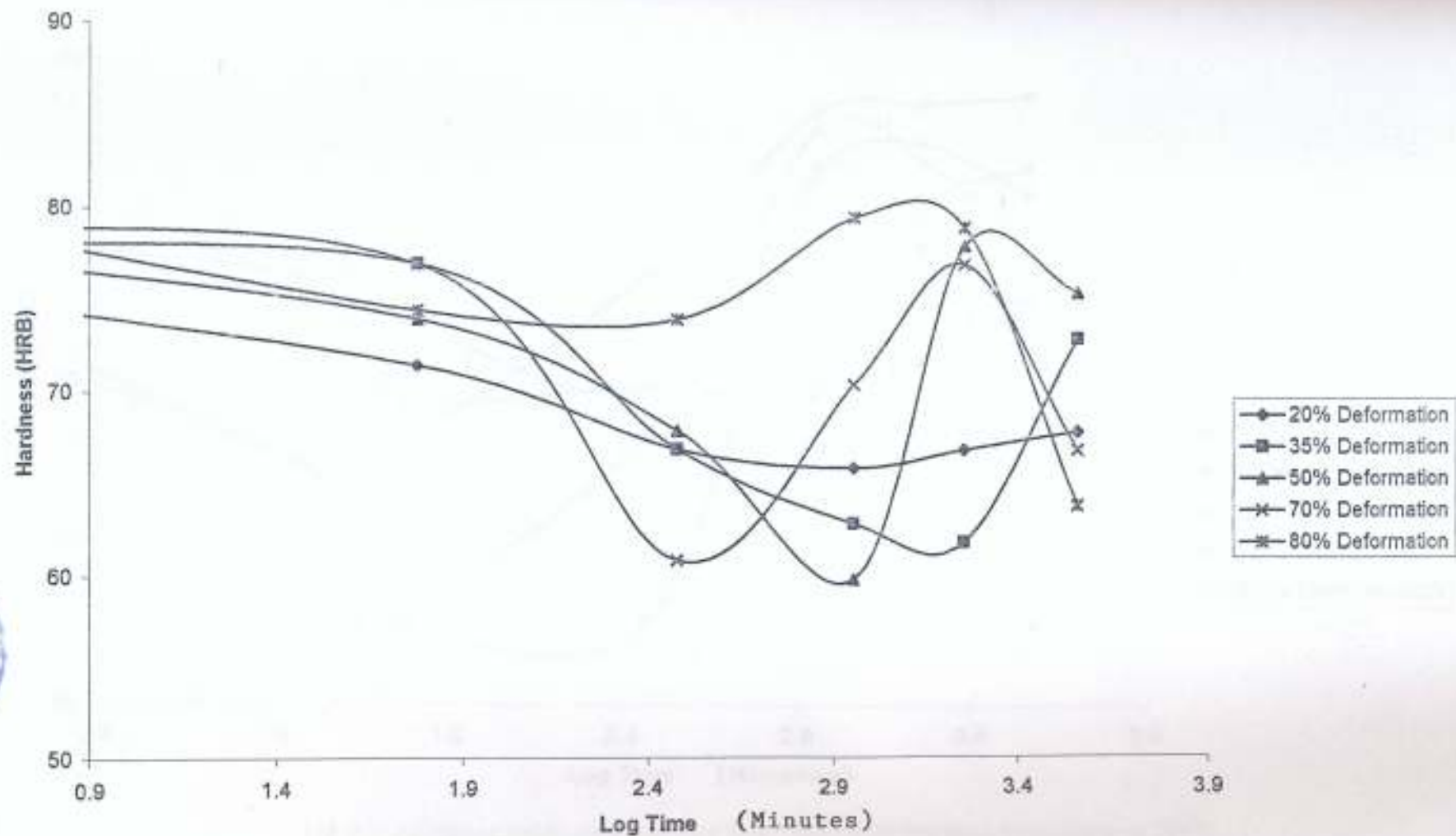


Fig. 4.1: Hardness (HRB) Vs Annealing Time Plot for Normalised Specimens at 740°C .

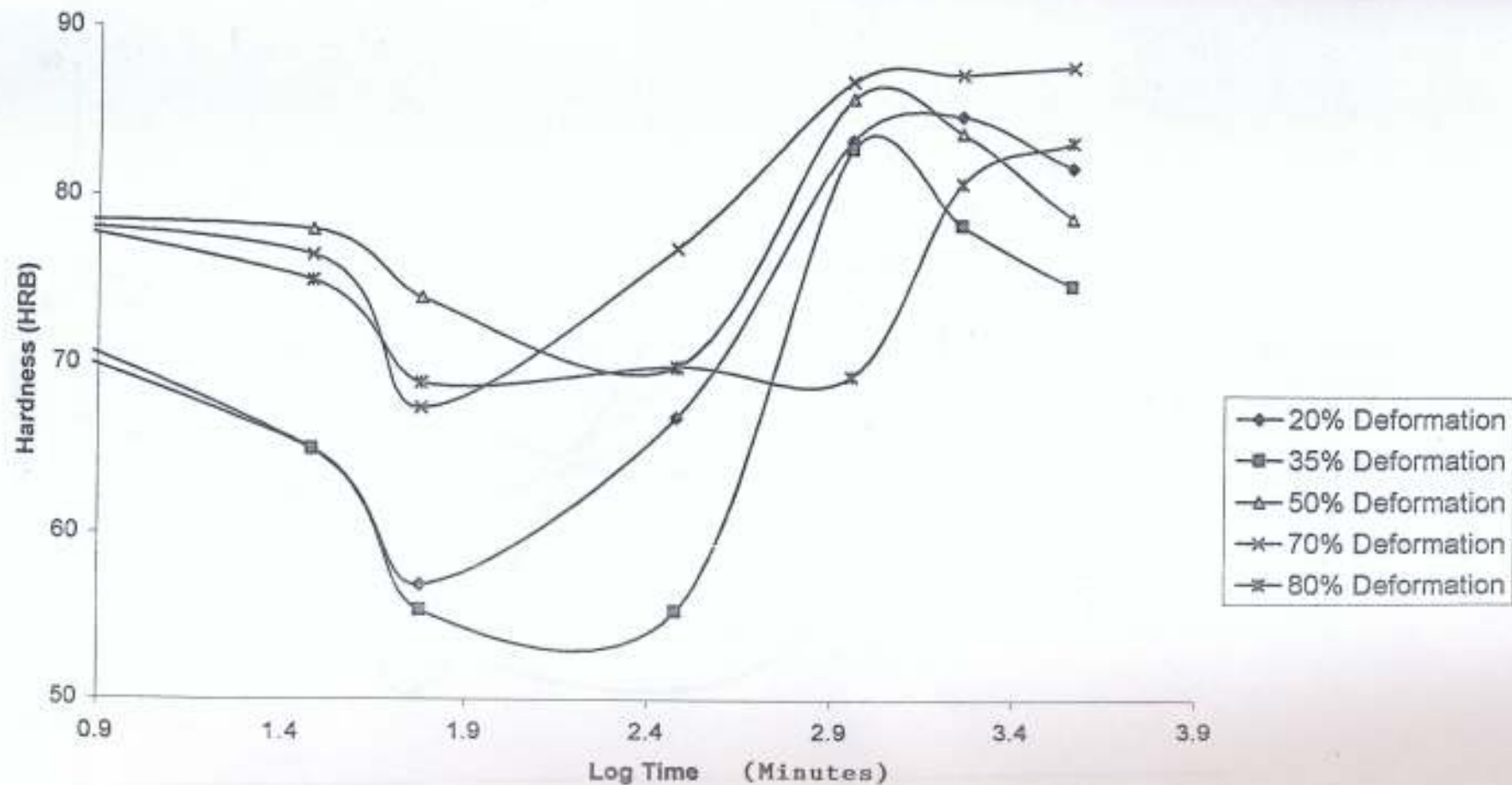


Fig. 4.2: Hardness (HRB) Vs Annealing Time Plot for Normalised Specimens at 760°C

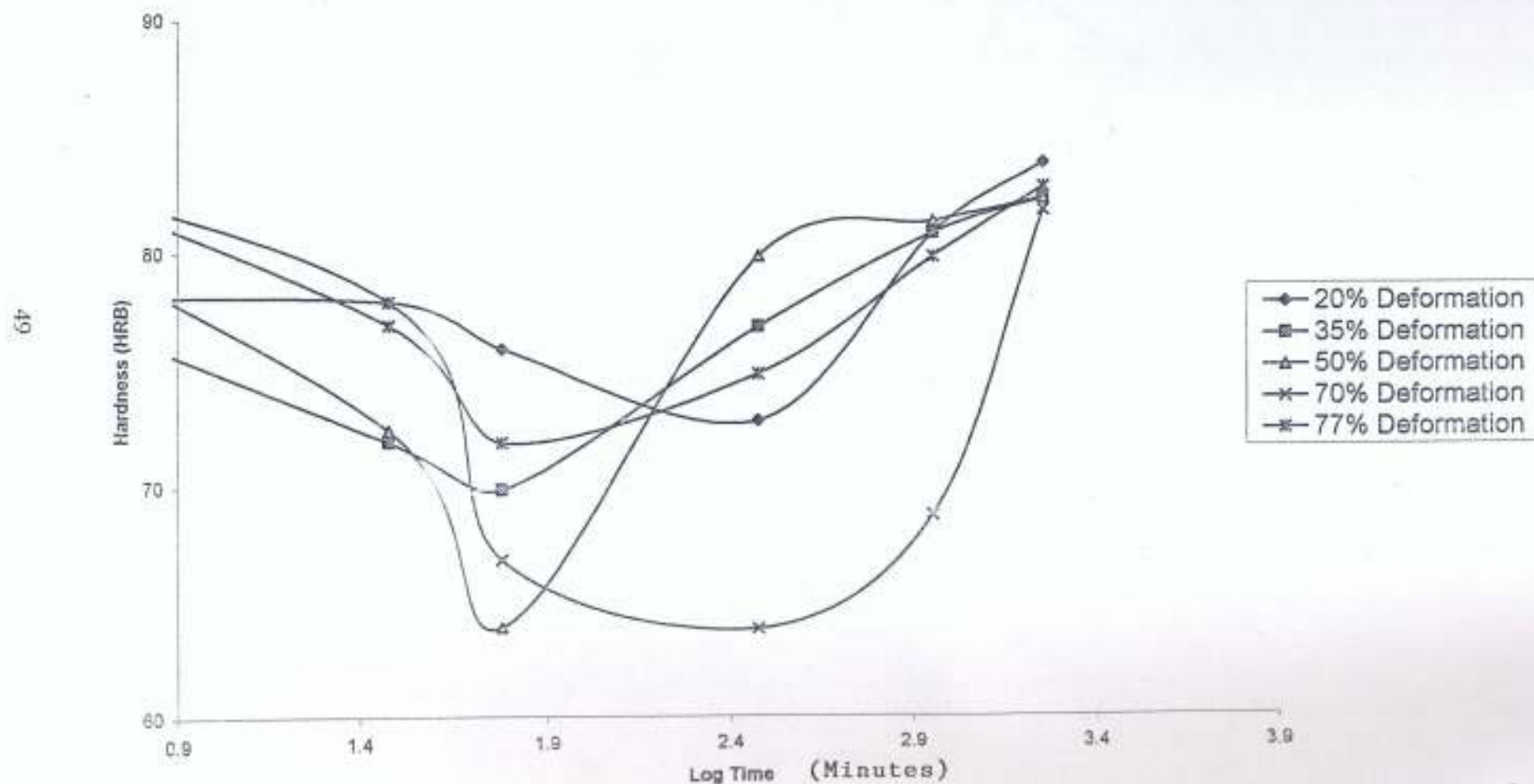


Fig. 4.3: Hardness (HRB) Vs Annealing Time Plot for Tempered Martensite Specimens at 740°C

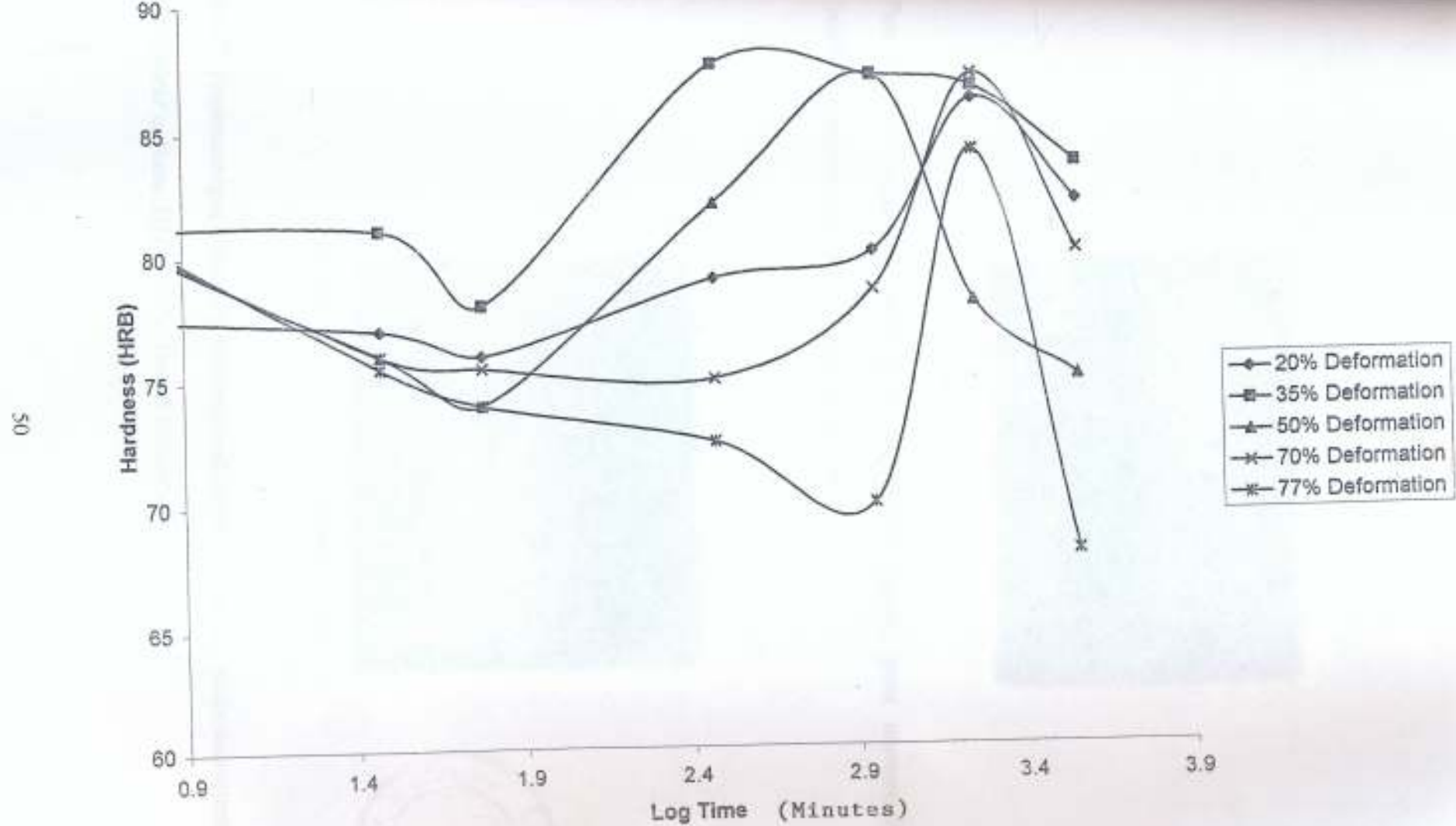


Fig. 4.4: Rockwell Hardness (HRB) Vs Annealing Time Plot for Tempered Martensite Specimens at 760°C.

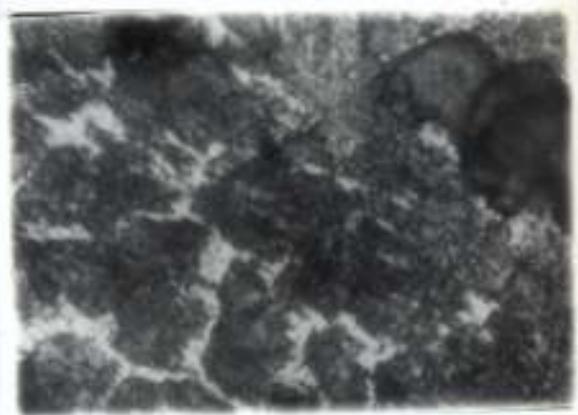


Plate 1: Photomicrograph showing the normalised starting point microstructure;
(pearlite/ferrite) structure 860°C, 1 hour/ air (400x)



Plate2: Photomicrograph showing the tempered martensite starting point microstructure;
(M450)860°C, 1hour/ H₂O +450°C, 1hour/H₂O(400x)

P/20%/740C, 0 – 1 hour (Plates 3 – 5): - The as – deformed microstructure (Plate 3) shows a divorced structure difficult to interpret. On annealing for 15minutes, it is observed from the microstructure shown in Plate 4 that pearlite coarsening (globularization) occurs; the coarse dark regions show pearlite. On heating for 1 hour, photomicrograph (Plate 5) shows incomplete recrystallization process.

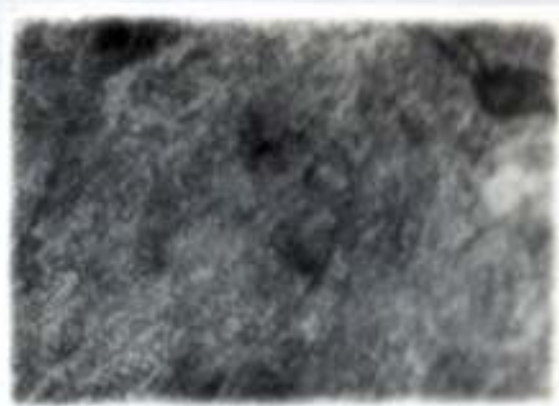


Plate 3: Photomicrograph showing the as-deformed structure of P/20%/740°C (400x).



Plate 4: Photomicrograph showing the microstructure of P/20%/740°C, 15mins/H₂O (400x)

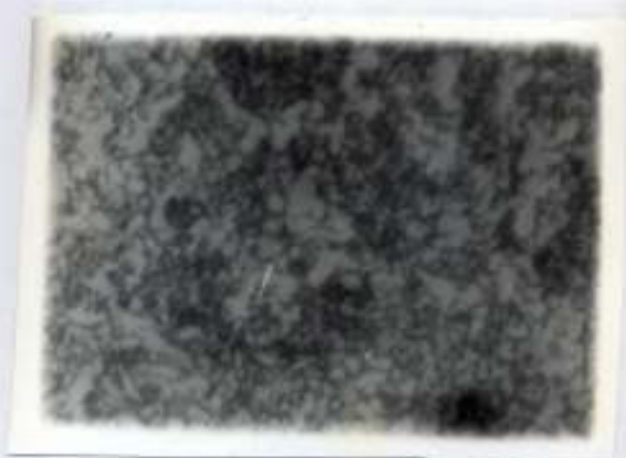


Plate 5: Photomicrograph showing the microstructure of P/20%/740°C, 1 hr/H₂O (400x)

P/50%/740°C, 0 – 1 hour (Plates 6 – 10) : - Plate 6 shows the as-deformed structure of the low carbon steel before annealing. On annealing for 1 minute (Plate 7), microstructure shows pearlite coarsening (globularization). After 15 minutes, (Plate 8) shows the onset of recrystallization as upper section of photomicrograph show small regions where recrystallization has started (white patches). On annealing for 30 minutes, microstructure consists of fine recrystallized grains (white patches) in martensite dark region (Plate 9). After 1 hour, microstructure shows coarse randomly distributed recrystallized grains (white) in martensite (grey) (Plate 10).



Plate 6: Photomicrograph showing the as-deformed microstructure of P/50%/740°C (400x)



Plate 7: Photomicrograph showing the microstructure of P/730%/740°C, 1' min/H₂O (400x).



Plate 8: Photomicrograph showing the microstructure of P/50%/740°C, 15mins/H₂O (400x)

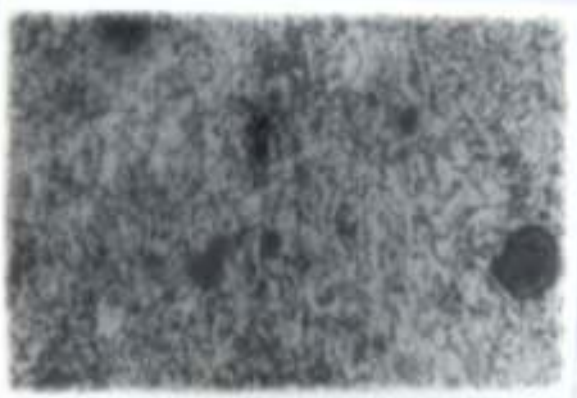


Plate 9: Photomicrograph showing the microstructure of P/50%/740°C, 30mins/H₂O (400x)

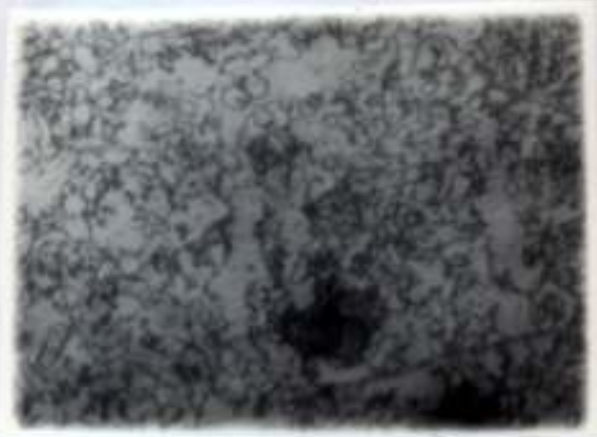


Plate 10: Photomicrograph showing the microstructure of P/50%/740°C, 1 hr./H₂O. (400x)

P/80%/740°C, 0 – 1 hour (Plates 11 – 14) : - The as-deformed structure is shown in Plate 11 which shows somewhat elongation of the pearlite (dark) and ferrite (white) structure in the direction of rolling. On annealing, rapid nucleation occurs after 5 minutes, as shown in Plate 12 which shows the onset of recrystallization as photomicrograph shows fine recrystallized nuclei in an untransformed pearlite matrix. Plate 14 shows a fairly coarse recrystallized grain (white) in a matrix of martensite after holding for 30 minutes.

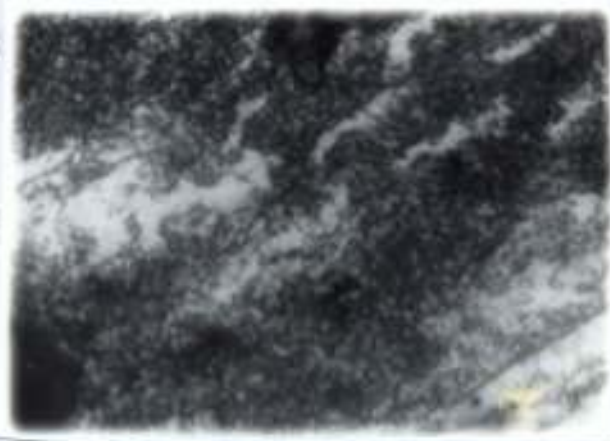


Plate 11: Photomicrograph showing the as-deformed microstructure of P/80%/740°C (400x).



Plate 12: Photomicrograph showing the microstructure of P/80%/740°C, 5mins/H₂O (400x)

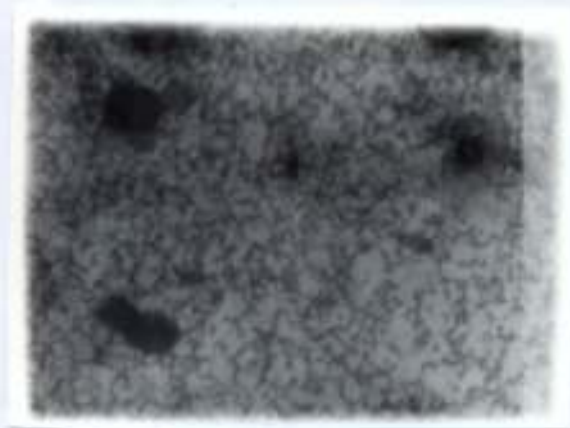


Plate 13: Photomicrograph showing the microstructure of P/80%/740°C, 15mins/H₂O (400x)

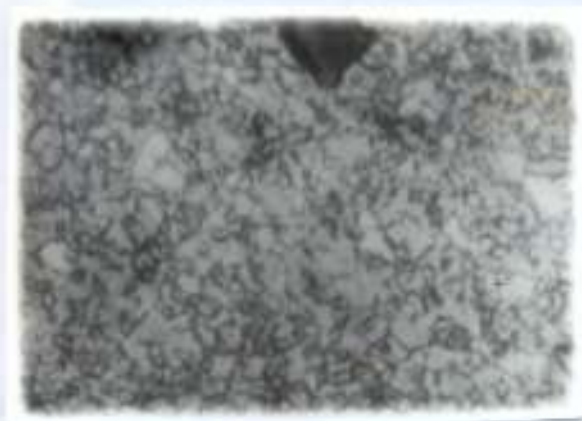


Plate 14: Photomicrograph showing the microstructure of P/80%/740°C, 30mins/H₂O (400x)

P/20%/760⁰C, 0 - 1hour (Plates 15 - 18): - The as deformed structure is shown in Plate 15, which is the same as Plate 3. On annealing, pearlite coarsening (globularisation) is observed as in annealing at 740⁰C (Plate 16). After 15 minutes an almost entirely acicular martensitic matrix with very few, fine and randomly distributed recrystallization nuclei is observed (Plates 17 and 18).

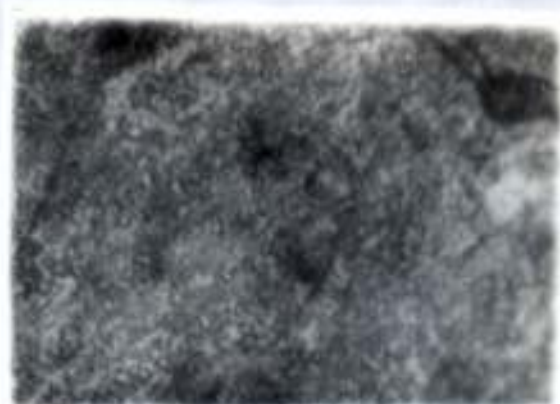


Plate 15: Photomicrograph showing the as-deformed microstructure of P/20%/760⁰C (400x).

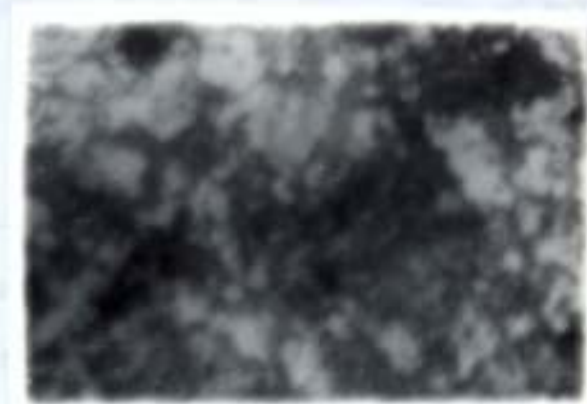


Plate 16: Photomicrograph showing the microstructure of P/20%/760⁰C, 5mins/H₂O (400x)

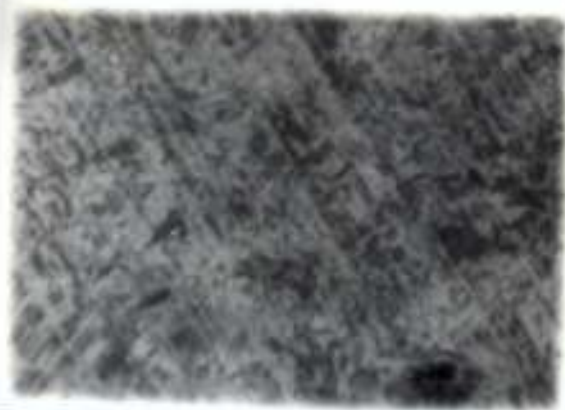


Plate 17: Photomicrograph showing the microstructure of P/20%/760°C, 15mins/H₂O (400x)



Plate 18: Photomicrograph showing the microstructure of P/20%/760°C, 30mins/H₂O (400x).

P/50%/760°C, 0 – 1 hour (Plates 19 – 23): - Plate 19 shows the as deformed structure. On annealing, pearlite globularization occurs as shown in Plate 20; after 5 minutes, microstructure gradually transforms to martensite as shown in Plate 21. Plate 22 shows that the microstructure after holding for 30 minutes, in this case a continuous network of martensite (dark) is seen surrounding the ferrite (white). On

holding for 1 hour, the photomicrograph (Plate 23) shows recrystallized grains in a martensite matrix.



Plate 19: Photomicrograph showing the as-deformed microstructure of P/50%/760°C (400x)



Plate 20: Photomicrograph showing the microstructure of P/50%/760°C, 5mins/H₂O (400x)



Plate 21: Photomicrograph showing the microstructure of P/50%/760°C, 15 mins/H₂O (400x)

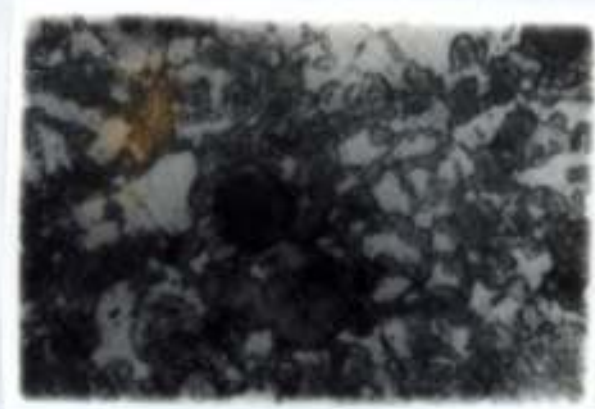


Plate 22: Photomicrograph showing the microstructure of P/50%/760°C, 30 mins/H₂O (400x)

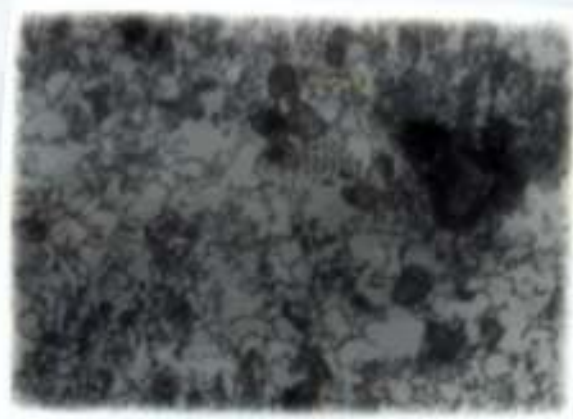
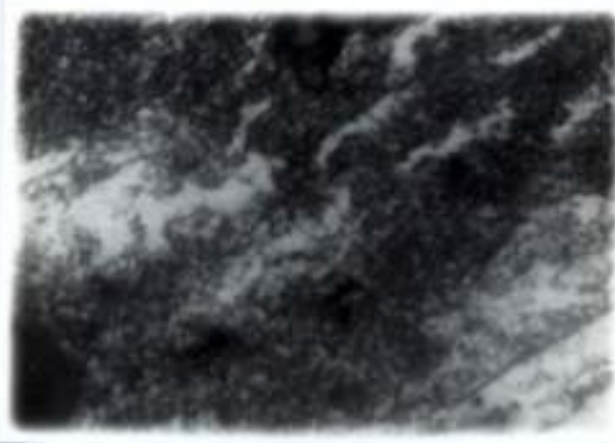
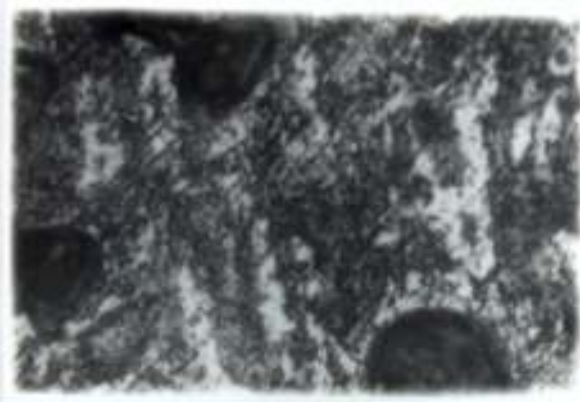


Plate 23: Photomicrograph showing the microstructure of P/50%/760°C, 1 hr/H₂O (400x)

P/80%/760°C, 0 – 1 hour (Plate 24 – 28): - Plate 24 shows the as deformed structure; Plate 25 shows pearlite globularization as in the case for 740°C on annealing for 1 minute. At 15 minutes annealing the recrystallization process is observed to have started as shown in Plate 26. Plate 27 shows the photomicrograph showing the advancement of the recrystallization front to the left side of the photomicrograph, and also well defined duplex structures of ferrite (white) and martensite (grey). On holding for 1 hour, microstructure shows well defined and fully formed duplex structure of ferrite (greyish white) and martensite (grey) (Plate 28).



24: Photomicrograph showing the as-deformed microstructure of P/80%/760°C,



te 25: Photomicrograph showing the microstructure of P/80%/760°C, 1 min/H₂O (400x)

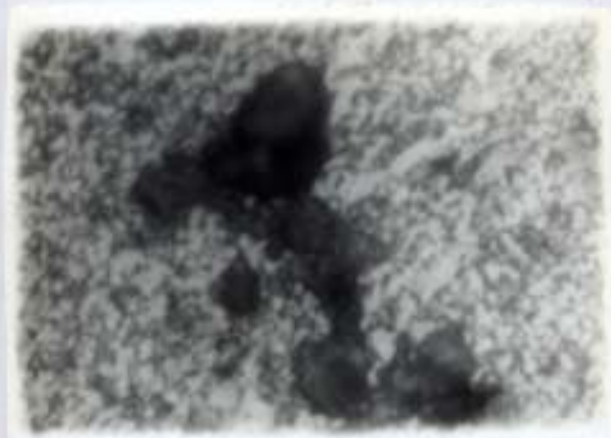


Plate 26: Photomicrograph showing the microstructure of P/80%/760°C, 15 mins/H₂O (400x)



Plate 27: Photomicrograph showing the microstructure of P/80%/760°C, 30mins/H₂O (100x)

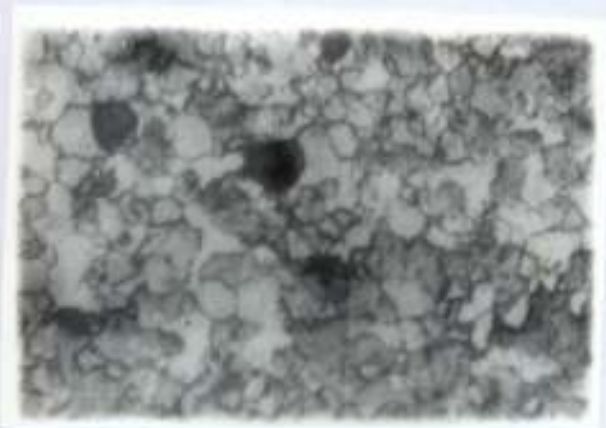


Plate 28: Photomicrograph showing the microstructure of P/80%/760°C, 1 hr/H₂O (400x)

M450/20%/740⁰C, 0 – 1 hour (Plates 29 – 33): - Plate 29 shows the as – deformed structure. On annealing for 5 minutes, the structure (Plate 30) shows a tempered martensite structure. At 15 minutes, it is observed that further precipitation of cementite particles occur which suppresses the growth of newly recrystallized grains; Plate 31 shows the microstructure after 15 minutes, which reveals a fine dispersion of cementite particles and very fine recrystallized grains. After 30 minutes, phase transformation (austenite precipitation) occurs alongside with recrystallization as Plate 32 and Plate 33 show fairly dispersed recrystallized grains in martensite.

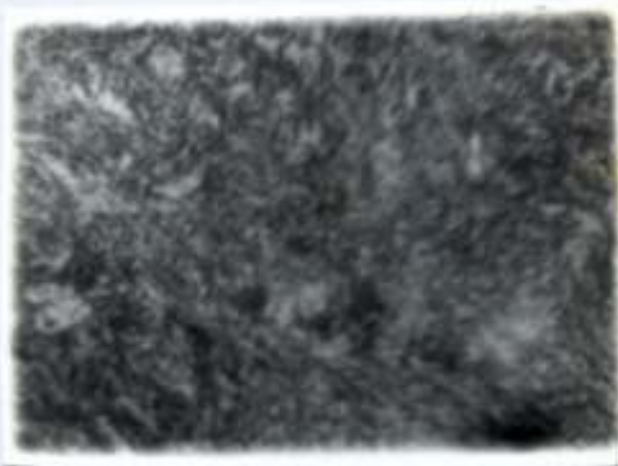


Plate 29: Photomicrograph showing the as-deformed microstructure of M450/20%/740⁰C (400x)



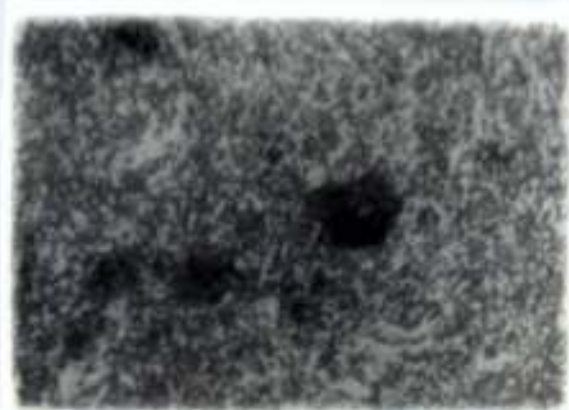
Plate 30: Photomicrograph showing the microstructure of M450/20%/740⁰C, 5 mins/H₂O (400x).

M450/50%/740°C, 0 – 1 hour (Plates 34-38): - The as – deformed structure (Plate 34) shows an elongated tempered martensite structure. On annealing for 5 minutes (Plate 35) the microstructure shows a tempered martensite structure, after 15 minutes (Plate 36) it is observed that the microstructure consist of recrystallized grains in martensite. After 15 minutes, the microstructure becomes greatly enriched by martensite as (Plate 37) shows a martensitic structure (dark) with few secondary recrystallized grains (big white) and fine recrystallized grains (small white). On annealing for 1 hour, the microstructure observed is martensite (dark) and recrystallized ferrite (white) (Plate 38).

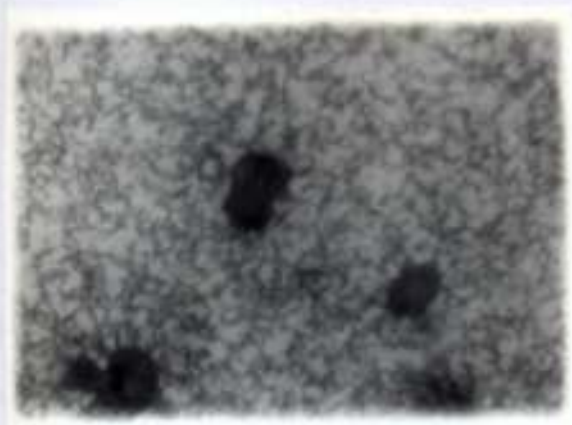


go to
after
plate
38

34: Photomicrograph showing the as-deformed microstructure of M450/50%/740°C (400x).



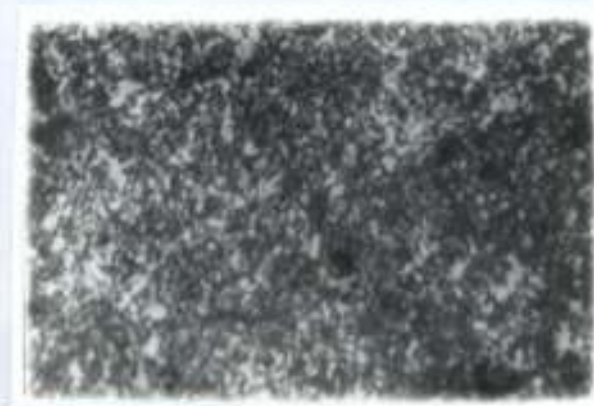
35: Photomicrograph showing the microstructure of M450/50%/740°C, 5 mins/H₂O (400x).



Photomicrograph showing the microstructure of M450/50%/740°C, 15 mins/H₂O (400X).



Photomicrograph showing the microstructure of M450/50%/740°C, 30 mins/H₂O



Photomicrograph showing the microstructure of M450/50%/740°C, 1 hr/H₂O (400x).

M450/77%/740°C, 0 – 1 hour (Plates 39-43): - Plate 39 shows elongated tempered martensite structure. On annealing, it is observed that until 15 minutes tempered martensite stress relief has occurred as Plates 40 and 41 show the growth of cementite particles. After 15 minutes, the microstructure shows fine recrystallized grains with cementite particles in martensite (Plate 42). The microstructure after 1 hour (Plate 43) shows fine recrystallized grains in martensite.



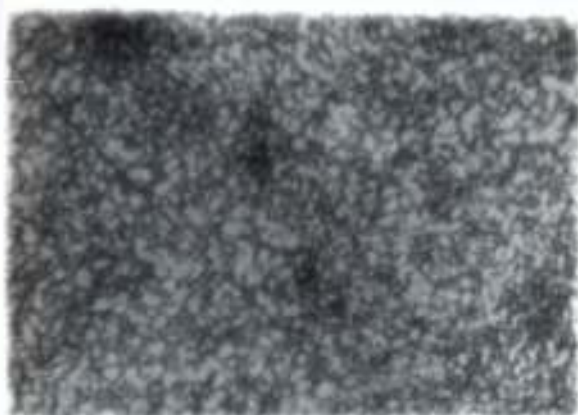
Plate 39: Photomicrograph showing the as-deformed microstructure of M450/77%/740°C (400x).



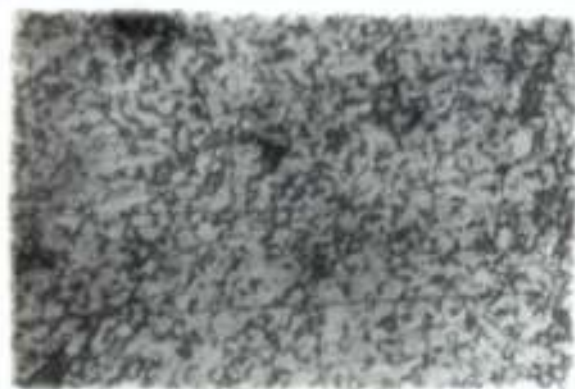
Plate 40: Photomicrograph showing the microstructure of M450/77%/740°C, 1 min/H₂O (400x).



41: Photomicrograph showing the microstructure of M450/77%/740°C, 5mins (400x).



42: Photomicrograph showing the microstructure of M450/77%/740°C, 15mins/H₂O (400x).



43: Photomicrograph showing the microstructure of M450/77%/740°C, 1hr/H₂O (400x).

M450/20%/760°C, 0 – 1 hour (Plates 44-47): -Plate 44 shows the as – deformed structure; on annealing for 5 minutes, the photomicrograph shows relief of stress as elongated structure has dispersed; also cementite particle growth and precipitation occur (Plate 45). On heating for 1 hour, microstructure shows primary and secondary recrystallized grains in martensite (Plate 47).



Plate 44: Photomicrograph showing the as-deformed microstructure of M450/20%/760°C (400x).

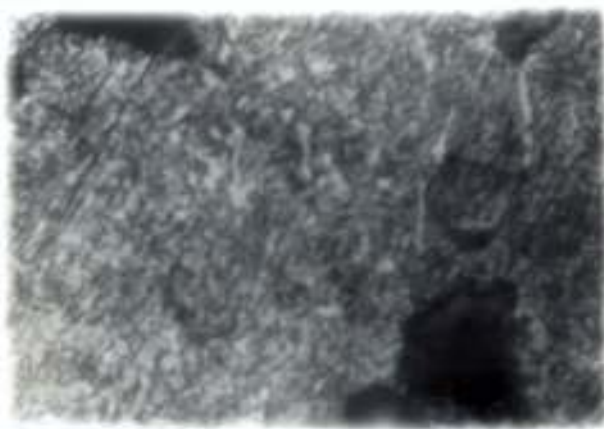
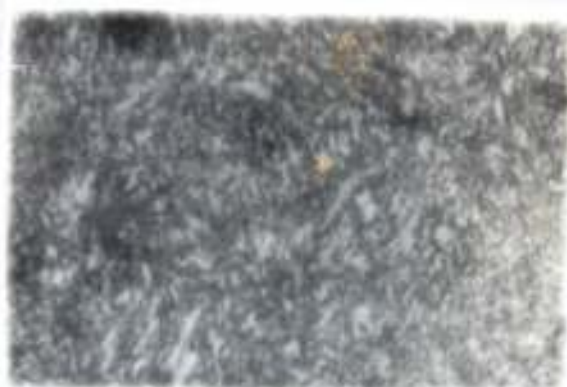
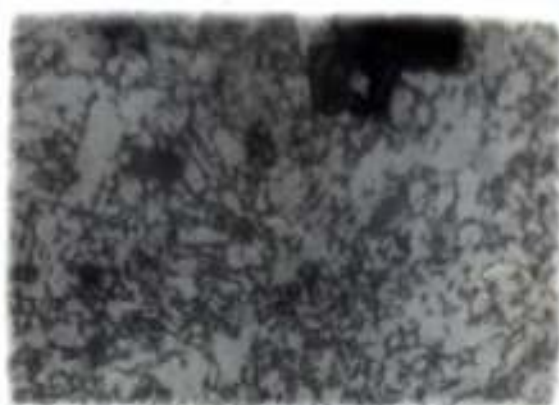


Plate 45: Photomicrograph showing the microstructure of M450/20%/760°C, 5 mins/H₂O (400x).



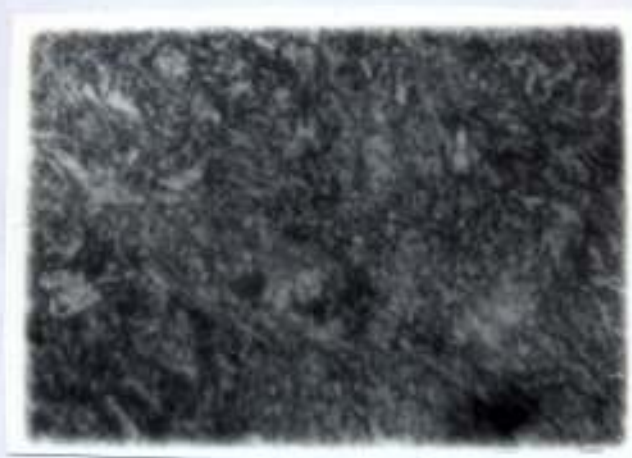
e 46: Photomicrograph showing the microstructure of M450/20%/760°C, 15 mins/H₂O (400x).



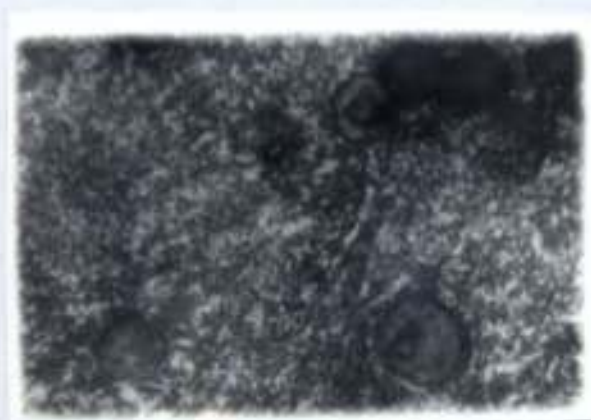
e 47: Photomicrograph showing the microstructure of M450/20%/760°C, 1 hr/H₂O (400x).

M450/50%/760°C, 0 – 1 hour. (Plates 48-51) :- The as-deformed structure is shown in Plate 48. On annealing for 1 minute, photomicrograph shows relief of stress as elongated structure has disappeared, also cementite particle growth and precipitation has occurred (Plate 49). The photomicrograph on annealing for 15 minutes shows precipitated cementite particles (black dots) in martensite (grey)

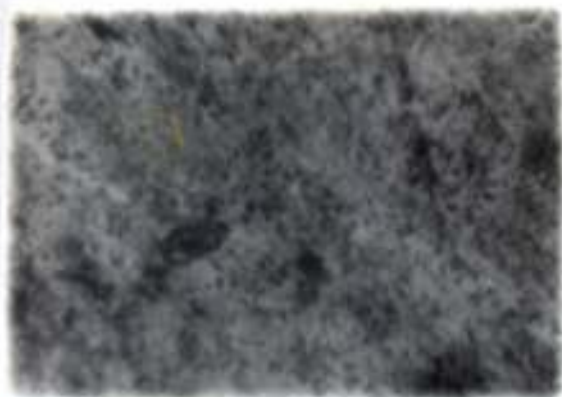
(Plate 50). On heating for 1 hour, microstructure shows primary and secondary recrystallized grains in martensite (Plate 51)



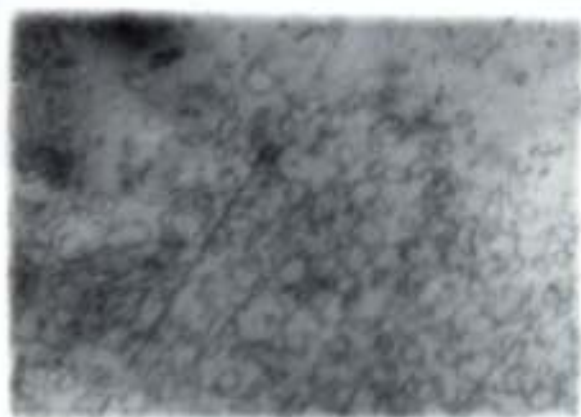
48: Photomicrograph showing the as-deformed microstructure of M450/50%/760°C,
(k).



49: Photomicrograph showing the microstructure of M450/50%/760°C, 1min/H₂O
(k).



50: Photomicrograph showing the microstructure of M450/50%/760°C, 15 mins/H₂O (400x).



51: Photomicrograph showing the microstructure of M450/50%/760°C, 1hr/H₂O (400x).

M450/77%/760°C, 0 – 1 hour (Plates 52-56):- The as-deformed structure is shown in Plate 52. On annealing for 1 minute, the microstructure is observed to have undergone stress relief that is tempered martensite stress relief (Plate 53). However on heating for 15 minutes, the microstructure shows that precipitation of cementite particles continues (Plate 54). At 30 minutes annealing, microstructure shows transformed martensite (Plate 55). After annealing for 1 hour, the

✓ go to after plate 56

microstructure is observed to have undergone recrystallization as structure consists of recrystallized grains in martensite (Plate 56).



2: Photomicrograph showing the as-deformed microstructure of M450/77%/760°C



3: Photomicrograph showing the microstructure of M450/77%/760°C, 1min/H₂O

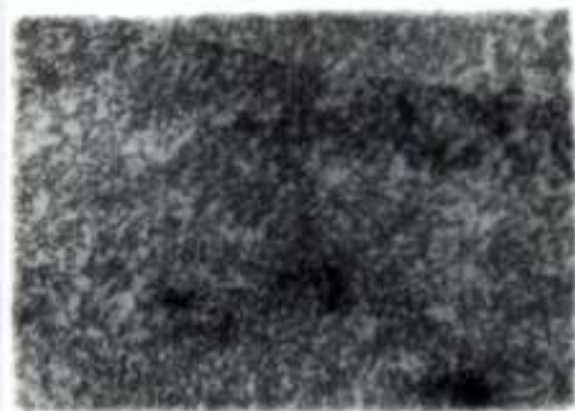


Plate 54: Photomicrograph showing the microstructure of M450/77%/760°C, 15mins/H₂O (400x).



Plate 55: Photomicrograph showing the microstructure of M450/77%/760°C, 30mins/H₂O (400x).

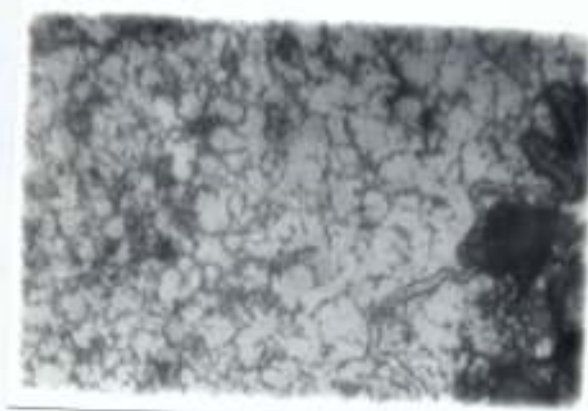


Plate 56: Photomicrograph showing the microstructure of M450/77%/760°C, 1hr/H₂O (400x).

4.1.4 Onset of Recrystallization:

Table 5 in appendix shows the results obtained from the determination of the onset of recrystallization for both starting point phases (that is, tempered martensite and normalized specimens) subjected to varied degrees of deformation before recrystallization annealing at 740°C and 760°C respectively.

4.2 DISCUSSION

In order to better understand and appreciate the factors influencing the recrystallization process in the ($\alpha+\gamma$) region observed here, an approach has been adopted which is deemed most suitable in discussing the obtained results. Firstly, proceeding from an examination of the microstructural conditions of each of the starting point microstructures before recrystallization annealing treatment. This is followed by a phenomenological study (descriptive analysis) of the combined effect of the various test parameters (initial microstructure, recrystallization annealing temperature, and degree of deformation) on the recrystallization process of each test specimen so as to determine the common trends of the recrystallization process, and hence establish a general mechanism for each of the two starting point phases. Finally, descending synthetically from the established general mechanisms to the individual effect of the test parameters on the recrystallization process so as to specify conditions under which the general mechanism proposed for each starting point phase would not be the norm.

4.2.1 Initial Microstructure

The pearlite (Plate 1) is an equilibrium structure that intrinsically can scarcely undergo further phase changes. Hence on prolonged heating at the intercritical temperature region, the only possible change that can occur before the commencement of ($\alpha \rightarrow \gamma$) transformation is globularization of the cementite in the pearlite. Higgins (1993), in his book said that prolonged heating at a higher temperature has an effect of causing excessive grain growth which leads to coalesce of the cementite in the patches of pearlite present leading to the final microstructure to be one consisting of globular masses of cementite in ferrite. Plate 4 and Plate 7 confirm this claim.

The tempered martensite structure in Plate 2, consists of cementite particles finely dispersed in the ferrite matrix. The M450 (tempered martensite structure) can be regarded as a quasi – stable structure, that is, capable of further precipitation on heating at a higher temperature. Thus during recrystallization annealing (which was done at a higher temperature), the precipitation process (cementite precipitation) that was incomplete in the course of the preliminary heat treatment for obtaining the starting point microstructure would continue before the commencement of phase transformation (austenite precipitation); also, the already precipitated cementite particles would coarsen (Kamma and Hornbogen, 1984). The photomicrograph, Plate 31 which is the microstructure after fifteen minutes recrystallization annealing when compared with Plate 29 which is the microstructure of the as – deformed test piece shows that further cementite precipitation occurs.

2.2 Interplay of Initial microstructure, Deformation and Temperature on recrystallization.

P/20%/740°C, 0-1 hour : - Using the hardness Vs annealing time plot in Fig 4.1 and the photomicrographs in Plates 3-5, it is observed that the as - deformed structure (Plate 3) experienced a gradual drop in hardness within the first fifteen minutes of recrystallization annealing. This fall in hardness is as a result of pearlite grain coarsening (globularization) and deformation stress relief (Higgins, 1993); this is shown clearly in Plate 4. On recrystallization annealing beyond fifteen minutes, a gradual rise in hardness is experienced as a result of gradual increase in the volume fraction of martensite, indicating that phase transformation has commenced. Yang and Chen (1991), have reported that increase in hardness is obtained on holding a low alloy steel for longer times as this will lead to an increase in the volume fraction of martensite. However, on close observation of the photomicrograph (Plate 5), it is seen that recrystallization has occurred, as the photomicrograph shows recrystallized grains (white grains). This implies that recrystallization occurs parallel with phase transformation (austenite nucleation). Kamina and Hornbogen (1984), have reported that recrystallization occurs as a result of the globularized cementite particles and former grain boundaries acting as sites for nucleation as a result of the adequate local lattice curvature usually associated with reduced energy barriers which they possess thus facilitating nucleation. Possible also, is the claim by Gorelik (1981), who reported that recrystallization can occur parallel with or immediately after phase transformation when there exist a volume differential between the old phase (ferrite, bcc) and the

nucleating new phase (austenite, fcc). This volume differential leads to structural strain-hardening, which engenders recrystallization.

P/50%/740°C. 0-1 hour : - In this case it is seen from fig. 4.1 that for the first fifteen minutes of recrystallization annealing, that a rapid fall in hardness is observed as a result of pearlite globularization and deformation stress relief (Higgins, 1993). Little or no recrystallization is observed in this time interval (Plate 7 and Plate 8). However, between fifteen and thirty minutes of recrystallization annealing, a sharp rise in hardness is observed as a result of increase in the volume fraction of martensite (Yang and Chen, 1991). But on close observation of the photomicrograph, Plate 9, which is the photomicrograph on annealing for 30 minutes, it can be inferred that recrystallization and phase transformation (austenite nucleation) occurred almost simultaneously. The effect observed has been explained by Gorelik (1981) who said that phase transformation associated with noticeable volume changes owing to difference between the unit volumes of old phase and the new phase (in this case bcc iron to fcc iron) leads to structural strain hardening which can result in recrystallization occurring after or parallel with phase transformation. The slight drop in hardness observed after 30 minutes is as a result of grain growth which in accordance with the Hall - Petch relationship

is associated with reduced number of grain boundaries which leads to decrease in mechanical properties (Hardness). Plate 10 shows that grain growth has occurred.

P/80%/740°C. 0-1 hour: - In this case, unlike the first two cases above, rapid nucleation of very fine recrystallized grains is observed before the

commencement of phase transformation (austenite precipitation), as can be seen in Plate 12 which is the photomicrograph on holding for five minutes. This rapid onset of recrystallization is associated with increased stored energy in the form of dislocations for nucleation of recrystallization nuclei, as dislocation density increases with degree of deformation (Haasen, 1997). However, the growth of the recrystallized grains triggers off the phase transformation process (austenite precipitation) as a rise in hardness is experienced after five minutes of annealing, signaling an increase in the volume fraction of martensite. Plate 13 and Plate 14 show that the recrystallization process occurs parallel with the phase transformation (austenite precipitation). According to Gorelik (1981), primary recrystallization can accelerate phase transformation (austenite precipitation) as a result of vacancies created during recrystallization. These vacancies accelerate the rate of diffusion of atoms thus helping to facilitate the process of dissolution of old phase (ferrite and cementite) towards the formation of a new phase (austenite).

P/20%/760°C, 0 – 1 hour: - This case unlike annealing at 740°C, experiences a sharper fall in hardness which is still due to pearlite globularization (Plate 16). However, after one minute of annealing, a sharp rise in hardness is observed as shown in Fig 4.2. This is as a result of increased volume fraction of martensite associated with phase transformation (austenite precipitation). The phase transformation (austenite precipitation) completely suppresses the recrystallization process as only very few recrystallized grains can be seen in Plate 17 and Plate 18. This acceleration of phase transformation (austenite precipitation) occurs in high strength low alloy steels at higher intercritical temperatures as a result of the easy

segregation of elements like Chromium, Manganese, and carbon whose atoms exhibit a form of "attractive Interaction" along grain boundaries. This attractive interaction will increase the rate of diffusion, and hence the rate of dissolution of ferrite and cementite to form austenite. Thus since this process predominates, then recrystallization would be greatly suppressed.

P/50%/760°C, 0 – 1 hour: In this case, phase transformation (austenite precipitation) occurs before recrystallization as Plate 20 and Plate 21 which are the photomicrographs on annealing for five minutes and fifteen minutes respectively show no sign of recrystallization but in fig. 4.2 this time interval signals somewhat the start and end of phase transformation (austenite precipitation). Even up to thirty minutes of annealing, there was still no sign of recrystallization as the photomicrograph (Plate 22) only shows a continuous network of globular martensite surrounding the ferrite matrix. But however, on annealing for one hour, recrystallization is observed to have occurred as shown in Plate 23. Again the initiation of phase transformation (austenite precipitation) and suppression of recrystallization is attributed to the "attractive interaction" between carbon, chromium, and manganese atoms which are easily segregated along grain boundaries during annealing at higher temperatures (Yang and Chen, 1991). Thus lower activation energy would be needed for phase transformation (austenite precipitation) to occur. However, the recrystallization that occurs after phase transformation (Plate 23) is attributed to structural strain hardening associated with volume change between the old phase (ferrite, bcc) and the new phase (austenite, fcc), which has the effect of initiating recrystallization (Gorelik, 1981).

P/80%/760°C, 0-1 hour: This case has a peculiar characteristic profile as shown in the hardness Vs annealing time plot in Fig. 4.2. It is seen that the initial fall in hardness is as a result of pearlite globularization, that there is mutual retardation of both the recrystallization and phase transformation (austenite precipitation) process as a plateau is observed in Fig. 4.2. Hornbogen and Koster (1978) have reported that the hindrance of the formation and migration of recrystallization by precipitates and the simultaneous promotion of precipitates by lattice defects, leads to both processes exerting mutual influence upon each other. Thus both processes try to suppress or prevent the other from occurring. However, on annealing for fifteen minutes, it is observed that recrystallization has commenced (Plate 26). On annealing further, it is observed that phase transformation (austenite precipitation) occurs in the recrystallized grains as revealed in Plate 27 and Plate 28 which shows recrystallized grains of ferrite (white) and martensite (grey). These are clear examples of duplex structures, which from grain size measurement, show that the P/80%/760°C,30mins/H₂O and P/80%/760°C,1hr/H₂O, are respectively 27.5µm and 10.6µm in size; and also were associated with increased hardness (strength) (Fig. 4.2). Hornbogen and Koster (1978) have reported that the temperature of annealing and defect concentration can have the effect of making normal recrystallization to occur before precipitation takes place in the recrystallized grains.

M450/20%/740°C,0-1 hour: - From Fig 4.3, it is seen that very slight drop in hardness is observed within the first five minutes of recrystallization annealing. This is as a result of further precipitation of cementite, which greatly retards the

recrystallization process as shown in Plate 31. Kamma and Hornbogen (1984), have reported that steels tempered under the same condition, undergo further precipitation which was incomplete during tempering when heated further at a higher temperature. This effect Gorelik (1981) has reported leads to retardation or complete suppression of the movement of dislocations and boundaries and thereby making the process of recrystallization to come to a halt. However, after fifteen minutes of annealing, a sharp rise in hardness is observed as phase transformation ensues; thus leading to increased volume fraction of martensite. The photomicrograph on recrystallization annealing for thirty minutes (Plate 32) shows clearly that the recrystallization process has been greatly retarded by the phase transformation (austenite precipitation) as only few randomly distributed recrystallized grains can be seen. However, on annealing for one hour (Plate 33), the number of recrystallized grains increases slightly, inferring that further recrystallization would take place in accordance with Gorelik (1981) who stated that structural strain hardening as a result of volume change between the old phase (ferrite, bcc) and the new phase (austenite, fcc) leads to primary recrystallization occurring immediately after or parallel to phase transformation.

M450/50%/740°C, 0-1 hour: - In this case, a sharp fall in hardness (Fig. 4.3) is observed within the first five minutes of annealing as a result of tempered martensite stress relief, and coarsening of precipitated cementite particles. After the cementite particles have reached a critical size, they serve as nucleation sites giving rise to accelerated recrystallization as shown in Plate 36. Kamma and Hornbogen (1984), have associated this acceleration of recrystallization to the fact that

inclusions (cementite) of critical size have adequate lattice curvatures with the attribute of reduced energy barriers thus making it possible for nucleation to occur at a faster rate. However, from the hardness Vs annealing time plot (Fig. 4.3), it is observed that the region in which recrystallization was observed annealing for 15 minutes) shown in Plate 36 coincides with an increase in the hardness, which is due to increase in the volume fraction of martensite. Thus implying that recrystallization occurred parallel with phase transformation (austenite precipitation). This as discussed earlier, Gorelik (1981) has attributed to structural strain hardening as a result of volume differential between the nucleating new phase (austenite, fcc) and the old phase (ferrite, bcc) which leads to recrystallization occurring parallel with phase transformation (austenite precipitation). On annealing further up to one hour, increase in the volume fraction of martensite, which Yang and Chen (1991) have said increases with holding time at a particular intercritical temperature.

M450/77%/740°C, 0-1 hour : - this case just as in M450/50%/740°C, there is a gradual fall in hardness (Fig. 4.3) within the first five minutes of annealing which is due to tempered martensite stress relief, and coarsening of precipitated cementite particles (Plate 40 and Plate 41). The cementite particles of critical size serve as nucleation sites for recrystallization but as can be seen in Plate 42 which is the photomicrograph of annealing for fifteen minutes, it is seen that there is suppressed growth of the finely recrystallized grains as a result of further precipitation of cementite particles and austenite nucleation. Hornbogen and Koster (1978) have reported that except when recrystallization is complete before

precipitate is nucleated, that the precipitate will exert a lot of influence on the recrystallization process as these precipitate particles hinder the formation and migration of recrystallization fronts thereby suppressing or halting the further growth of the recrystallized grains. On annealing for one hour, it can be seen from Plate 43 that there is complete suppression of the recrystallization process. The increased hardness is associated with increase in the volume fraction of martensite.

M450/20%/760,0-1 hour: - From the hardness Vs annealing time plot in Fig 4.4, it is seen that a very slight fall in hardness is witnessed within the first five minutes before a gradual rise in hardness occurs. The fall in hardness is attributed to tempered martensite stress relief and cementite particle growth as earlier discussed (Plate 45). After one minute the gradual rise is as a result of martensite volume enrichment (Yang and Chen, 1991), implying that the precipitation process starts before and completely suppresses the recrystallization process (Plate 46). Yang and Chen (1991), have attributed the case of nucleation of austenite on annealing at higher intercritical temperatures to the fact that the attractive interaction between chromium, carbon, and manganese atoms already segregated at the grain boundaries facilitate the diffusion of carbon atoms thus enhances austenite nucleation. However, after annealing for above thirty minutes, primary recrystallization occurs but its growth is greatly hindered by the phase transformation process (austenite precipitation). Plate 47 shows fine recrystallized grains in the martensitic matrix. As earlier discussed, Gorelik (1981), has attributed this effect to structural strain hardening arising as a result of volume difference

between the new phase (austenite) and the old-phase (ferrite). On heating to one hour, fall in hardness is witnessed as a result of grain growth.

M450/50%/760°C, 0-1 hour: This follows the same trend as M450/20%/760°C; there is initially a slight drop in hardness within the first one minute of annealing as shown in Fig 4.4, as before the reason is as a result of tempered martensite stress relief and cementite particle growth. After one minute a sharp rise in hardness is observed as a result of increasing volume fraction of martensite. The microstructure at holding for fifteen minutes (Plate 50), shows no sign of recrystallization. This shows that the phase transformation process completely takes pre-eminence as the recrystallization process is completely suppressed. However, on annealing for one hour, a sharp fall in hardness is observed as a result of recrystallization process, which has commenced. Gorelik (1981) has attributed this to structural strain hardening effect, which engenders recrystallization.

M450/77%/760°C, 0 - 1hour: In this case, a slightly different trend is observed. It starts initially with a fall in hardness, which is as a result of tempered martensite stress relief and cementite particle growth within the first thirty seconds. However, after this time lapse very little drop in hardness is observed as both the recrystallization and phase transformation (austenite precipitation) processes are completely inhibited. Kamma and Hornbogen (1984), have attributed this effect to the broadening of the deformation bands and with the help of the fine dispersion of the precipitated cementite particles lead to homogenization of strain resulting in the formation of fewer recrystallization nuclei and overall delay of the recrystallization

process. Thus, the incubation period for recrystallization and phase transformation (austenite precipitation) to occur is greatly increased. However, between fifteen minutes to thirty minutes, a sharp rise in hardness is observed as shown in Fig. 4.4, as a result of increasing volume fraction of martensite due to austenite precipitation. After thirty minutes, drop in hardness is witnessed as combination of cementite particle growth (Kamma and Hornbogen, 1984) and structural strain hardening (Gorelik, 1981) lead to nucleation and growth of recrystallized grains hence fall in hardness. Plate 56 shows randomly recrystallized grains in martensite on annealing for one hour.

Influence of temperature on Recrystallization Kinetics.

From the summary chart on table 4.5 in appendix, it is seen that there is a general delay or suppression of the onset of recrystallization annealing at 760°C ; while the incubation period for the initiation of austenite precipitation was shorter at 760°C in comparison with recrystallization annealing at 740°C . Yang and Chen (1991), have reported that when annealing in the $(\alpha+\gamma)$ region, that austenite nucleation at the prior austenite boundaries is greatly enhanced by the attractive interaction between carbon, chromium and manganese atoms already present at the boundaries if the annealing temperature is high. Thus the diffusion process necessary for the formation of austenite nuclei will be greatly facilitated. This implies that the overall activation energy required for austenite nucleation at 760°C is lower than that for recrystallization, and thus austenite precipitation precedes and controls the rate of recrystallization. Thus there is need to introduce a new criterion

to the activation energy model for phase transformation proposed by Hornbogen and Korster (1978). Hornbogen and Korster (1978) proposed that the time for the beginning of transformation, t_p be expressed as.

$$t_p = k_p \exp [(Q_n + Q_D)/kT] \quad (\text{recall equation ix})$$

Where k_p = transformation constant

Q_n = activation energy for nucleation of new phase

Q_D = activation energy for the diffusion of foreign atoms.

K = Boltzmann constant

T = Absolute temperature

But the contribution of the attractive interaction between carbon, chromium, and manganese atoms to the lowering of the activation energy necessary for phase transformation, will necessitate the introduction of an activation energy criterion, Q_A , which should be subtracted from $(Q_n + Q_D)$. Thus t_p should be expressed as :

$$T_p = k_p \exp [\{ (Q_n + Q_D) - Q_A \} / KT] \quad \dots\dots\dots (x)$$

Where Q_A = energy contribution by attractive interaction of carbon, chromium, and Manganese atoms towards the promotion of carbon diffusion and thus lowering of the overall activation energy required for formation of austenite nuclei.

4.2.4 Influence of Deformation on Recrystallization Kinetics.

From table 4.5 in appendix, it is seen that for the normalized initial microstructure, P , the time for the initiation of recrystallization decreases with increasing deformation. This is due to the fact that higher degree of deformation

increases the severity of lattice curvature, making it possible for a small growing subgrain to easily acquire a large angle boundary and thus become an active nucleus for recrystallization (Smallman, 1992). Haasen (1997) has associated higher degree of deformation with increase in the stored energy in form of dislocations necessary to overcome the barriers for recrystallization. Thus the larger the deformation, the greater the tendency for recrystallization to occur, and hence the faster the rate of recrystallization. However, for the initially tempered starting point microstructure, M450, it is observed that at deformation, $E=77\%$, at 740°C , the recrystallization process was delayed (took longer time) in comparison with deformation at $E = 20\%$ and $E = 50\%$, at 740°C . This is as a result of broadening of the deformation bands which leads to the homogenization of strain, resulting in the formation of fewer nuclei and delayed progress of recrystallization (Kamma and Hornbogen, 1984). This in addition to the influence of austenite precipitation contributes to the delay in the initiation of recrystallization process.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In a bid to investigate the technical viability of alloy development via thermomechanical processing (recrystallization annealing), the recrystallization kinetics of a 0.4C-1.19Mn-0.26Si-0.13Cr alloy steel subjected to recrystallization annealing in the ($\alpha + \gamma$) phase transformation region was studied. Two phases tempered martensite (M450) and pearlite/ferrite (P) were prepared as starting point phases (initial microstructures), before being subjected to various degrees of cold rolling. The cold rolled samples were subsequently subjected to recrystallization annealing at 740°C and 760°C at various times ranging between 30 seconds to 60 minutes. From the resulting microstructure and hardness measurements, the following conclusions are drawn:

1. Recrystallization annealing at 740°C, was observed to lead to recrystallization and austenite precipitation occurring parallel to each other for both starting point microstructures; while recrystallization annealing at 760°C led to suppression of the recrystallization process, in favour austenite precipitation: especially in the tempered martensite starting point phase specimens.
2. Well defined duplex structures of recrystallized ferrite and martensite can be produced in the low alloy steel, by using a pearlite/ferrite starting point

structure, subjected to 80% deformation, before annealing at 760°C for 30 minutes or 1 hour, and then quenched in water: in which the duplex structure obtained would have average grain size of 27.5µm or 10.6µm respectively.

3. As the degree of deformation increased, the rate of recrystallization also increased for the normalized specimens, when they are annealed at 740°C. This was however, not the case for the tempered specimens, in which retardation of the recrystallization kinetics was observed when the degree of deformation was increased to 77%.
4. Recrystallization in the ($\alpha + \gamma$) transformation region was for most cases associated with hardening of the low alloy carbon steel.
5. The influence of the attractive interaction between chromium, manganese, and carbon atoms, towards facilitating the austenite precipitation process and thus reducing the overall activation energy requirement for nucleation, necessitates the modification of the Arrhenius type equation for the onset of the phase transformation (austenite precipitation) for the 0.4C-1.19Mn-0.26Si-0.13Cr alloy steel to

$$t_p = K_p \exp[(Q_n + Q_d) - Q_A]/KT$$

where, Q_A = energy contribution by attractive interaction of carbon, manganese, and chromium atoms towards the reduction of the overall activation energy required for nucleation of austenite.

5.1 Recommendation

Further research efforts should be centred on the mechanical properties of the different duplex microstructures produced (recrystallized ferrite in martensite) so as to determine the possible limitations to their use. There should also be efforts in studying the mechanism of the recrystallization process in the $(\alpha+\gamma)$ phase transformation region of the low alloy steel with the aid of electron microscope. This would enable changes in the substructure of the low alloy steel as a result of recrystallization/austenite precipitation mutual interaction to be properly studied.

REFERENCES

- Ajayi, J. A. (1997); Strain Hardening Characteristics of NS34LC, NSE Technical Transactions, Vol. 32, No. 3, pp 50 – 62.
- Gorelik, S. S. (1981); Recrystallization in metals and alloys, 2nd Edition, Mir Publishers: Moscow.
- Haasen, P. (1997); Physical Metallurgy, 3rd Edition; Cambridge: Great Britain.
- Hansen, N. (2001); New Discoveries in Deformed Metals, Metall. Mater. Trans., Vol 32 A, No 12, pp 2917 – 2919.
- Higgins, A. R. (1993); Engineering Metallurgy Part I: Applied Physical Metallurgy, 6th Edition; Edward Arnold: England.
- HoneyCombe, R. W. K. (1987); Steel Microstructure, Edward Arnold (Publishers): London.
- Hornbogen, E. and Koster, U. (1978); Recrystallization of Metallic Materials (Ed. Haessner, F.) Riederer, Stuttgart, pp 159.
- Humphreys, F. J. and Hatherly, M. (1995); Recrystallization and Related Annealing Phenomena, Elsevier Science, Oxford: U.K, pp 3-423.
- Kamma, C. M. (1990); Optimum Conditions for Grain Refinement in Carbon Steel by Thermomechanical Treatment, Materials Forum, 14, pp 62 – 66.
- Kamma, C. M. (1993); Design of Microstructures in Mild Steel by Thermomechanical Treatment, Proc. Of N.M.S., 11, pp 8 – 18.
- Kamma, C. M. and Hornbogen, E. (1984); Recrystallization Mechanism in Carbon Steel, Canad. Met. Quarterly, Vol 23, No 2, pp 249 – 257.

- Martin, J. W., Doherty R. D and Cantor B (1997); *Stability of Microstructure in Metallic Systems*; 2nd Edition; Cambridge University Press: UK.
- Porter, D. A. and Easterling, K. E., (1993); *Phase Transformation in Metals and Alloys*, 2nd Edition, Chapman and Hall: London.
- Raghavan, V. (1989); *Physical Metallurgy: Principles and Practice*, Prentice-Hall of India: New Delhi.
- Rajan, T. V., Sharma, C. P and Sharma, A. (1988); *Heat treatment Principles and Techniques*, Prentice Hall of India; New Delhi.
- Smallman, R.E. (1992); *Modern Physical Metallurgy*, 4th Edition, Butterworth-Heinemann Ltd: London.
- Torres, R.C.E, Sanchez, F. H., Gonzalez, A., Actis F., and Herrera, R. (2002); Study of the Kinetics of the Recrystallization of Cold-Rolled Low Carbon Steel, *Metall. Mater. Trans. Vol. 32 A*, No 1, pp 25 – 31.
- Avrami, L. H. (1970); *Materials Science For Engineers*, Addison-Wesley Publishers: Massachusetts.
- Giaoyan, S., Markus R, Clemens M and Hans E. (2001); Modelling of Recrystallization after Inhomogenous Deformation, *Metall. Mater. Trans. Vol 32 A*, No 9, pp 2199 – 2206.
- Yang, J. R. and Chen, L. J. (1991); Dual Ferrite-Martensite Treatments of a High-Strength Low-alloy ASTM A588 steel. *Journal of Materials Science*, 26, pp 889 – 898.

APPENDIX

Table 1: Rockwell Hardness numbers for normalised starting point specimens with annealing time at 740°C.

Deformation %	Rockwell Hardness Number (HRB) 100kgf with time					
	0 sec	1 minute	5 minutes	15 minutes	30 minutes	1 hour
20	76.5	71.5	67	66	67	68
35	78	77	67	63	62	73
50	78.5	74	68	60	78	75.5
70	79	77	61	70.5	77	67
80	81	74.5	74	79.5	79	64

Table 2: Rockwell Hardness number for normalised starting point specimens with annealing time at 760°C

Deformation %	Rockwell Hardness Number (HRB) 100kgf with time						
	0 sec	30 secs	1 minutes	5 minutes	15 minutes	30 minutes	1 hour
20	76.5	65	57	67	83.5	85	82
35	78	65	55.5	55.5	83	78.5	75
50	78.5	78	74	70	86	84	79
70	79	76.5	67.5	77	87	87.5	88
80	81	75	69	70	69.5	81	83.5



Table 3: Rockwell Hardness number for tempered martensite starting point specimens with annealing time at 740°C

Deformation %	Rockwell Hardness Number (HRB) 100kgf with time					
€	0 sec	1 minutes	5 minutes	15 minutes	30 minutes	1hour
20	78	78	76	73	81	84
35	81	72	70	77	81	82.5
50	85	72.5	64	80	81.5	82.5
70	85.5	78	67	64	69	82
80	86.5	77	72	75	80	83

Table 4: Rockwell Hardness number for tempered martensite starting point specimens with annealing time at 760°C

Deformation %	Rockwell Hardness Number (HRB) 100kgf with time						
€	0 sec	30secs	1minutes	5minutes	15minutes	30minutes	1hour
20	78	77	76	79	80	86	82
35	81	81	78	87.5	87	86.5	83.5
50	85	76	74	82	87	78	75
70	85.5	76	75.5	75	78.5	87	80
80	86.5	75.5	74	72.5	70	84	68

Table 5: Quasi Kinetics Analysis Chart

Sample	Coldwork	740°C			760°C		
	c (%)	T_{pb}	t_{PE}	t_{RI}	T_{pb}	t_{PE}	t_{RI}
	0	-	-	-	-	-	-
P	20	≥ 15 mins	> 1 hr	≥ 15 mins < 30 mins	1min	30mins	Suppressed within 1hr.
	50	15mins	30mins	15mins	5mins	15mins	> 30 mins < 1 hr
	80	≥ 15 mins	> 1 hr	5mins	15mins	> 1 hr	> 1 min < 15 mins
	0	-	-	-	-	-	-
M450	20	15mins	> 1 hr	5mins	1min	30mins	> 15 mins < 30 mins
	50	5mins	> 1 hr	< 5 mins	1min	15mins	> 15 mins < 1 hr
	77	5mins	> 1 hr	15mins	15mins	30mins	> 30 mins < 1 hr

Note:

 t_{RI} = Time for initiation of recrystallization T_{pb} = Precipitation of Austenite begins t_{PE} = Precipitation of austenite ends