CHAPTER I
Introduction

1.1 Changes in the Quality of Steel

Of all materials, steel is unique because of its certain qualities. It possesses a combination of strength, toughness, rigidity, workability and low cost. Its resources are abundant and field of application is wide. The scale of world’s steel production, which is around 700 million tons per year, testifies this\(^1\).

The properties and performance capabilities of steels have undergone significant changes over the years to meet the demands of the industries and the society. Because of rising energy costs, rigorous service conditions, stringent consumer preferences and ecology awareness, today it has become necessary to produce steels of higher quality with advanced functions at lower cost.

Concurrent with the changing needs of users, the steel making and processing technologies have undergone significant changes. The aim is now to produce clean steels with enhanced product qualities and properties. This is possible through right choice of alloy chemistry, production and processing conditions. This results in the right combination of cleanliness, grain size and microstructure which impart the desired end-properties in the steel.

1.2 Emerging Trends in High Strength Steels

The need for high strength steels with higher strength-to-weight ratio is steadily increasing for stronger, larger, durable and economic structures. The class
of high strength steels is normally categorised by their yield strengths ranging from 350 to 800 MPa. Traditionally the strength levels of hot-rolled steels were increased through addition of higher levels of carbon and manganese. Though this resulted in achieving higher strength levels, it seriously affected ductility, low temperature toughness and weldability properties. On the other hand, Q&T steels derived its strength from carbon level, alloy additions and the heat treatment process. The alloy additions imparted hardenability through a martensitic structure, which is subsequently tempered to achieve the desired combination of strength, toughness and ductility. While the base metal properties of this class of steel are sufficient, it suffers from weldability problems.

The introduction of thermomechanically controlled processing (TMCP) in the early seventies has revolutionised the art of high strength steel production. The stage-wise evolution of this process is a great success story and is given in Chapter II (Art.2.8.2). It is now possible, through controlled deformation of the austenite in the hot rolling stage and subsequent transformation during cooling, to achieve a fine grain size with desired microstructure.

The critical features of a TMCP process are: suitable selection of alloy chemistry; choice of slab reheating temperature; degree of deformation in the γ-recrystallization and γ-non-recrystallization zones and finally controlled cooling. The slab reheating temperature determines the initial grain size and distribution of micro-alloyed elements in the form of solid solution or precipitates. Adequate deformation is imparted in each pass of primary rolling (γ-recrystallization zone) to achieve repeated recrystallization of the austenite. Further down, the reduction in the finish rolling stage (γ-non-recrystallization zone) further refines the austenite grain size. Controlled cooling after finish rolling helps in attaining a wide array of microstructure from ferritic-pearlitic to acicular ferrite/bainite structure.
The requirement for steels with higher strength with enhanced toughness and weldability has prompted intensive examination of non-equiaxed ferrite microstructures in low carbon and ultra low carbon steels. By virtue of a low carbon content and low carbon equivalent, these steels exhibit lower susceptibility to weld cracks during sudden dynamic cyclic loading. At the same time the yield ratio (YS/TS), an important factor in design, is above 80% which ensures adequate energy absorption capability.

1.3 Options and Opportunities

The options and opportunities before the steel industry are enormous because of the availability of powerful process technologies in modern steel mills. Though TMCP technology has revolutionised steel production, a deeper understanding of the deformation process, especially in multi-pass rolling, is required to exploit this technology further. Developments of microstructure models are necessary for predicting the changes in microstructure and the associated properties of steels which have been control rolled and control cooled. This will lead to development of steels with tailor-made properties using a single alloy chemistry by manipulating the process parameters. This will solve many logistic problems in production while satisfying the need of small lot supply of tailor-made steels.

1.4 Statistical Design of Experiments

By the application of statistical design of experiments it is possible to develop an alloy with optimal combination of properties of an alloy by carrying out a limited number of experiments. Together with simultaneous variation of all
the parameters and applying mathematical analysis, the optimum solution can be guaranteed. The approach of generating the formal mathematical relationship in the form of equations to obtain optimum solution may not directly focus on the physical understanding of the mechanisms involved. However, knowledge of the nature of variation of the regression coefficients complemented by classical tools of research (like SEM, TEM etc.) help in understanding the mechanism involved.

The method works on the principle of Black Box, where a purely formal relationship among the inputs and the output is brought out with the help of mathematical statistics. Under the mathematical model it is understood that there exists a system of equations expressing relationships among the inputs (various processing parameters) and the output or response (property of a steel). The relation can be expressed in the form of an equation:

\[ Y = \varphi (X_1, X_2, ..., X_n) \]  
\[ \text{(1.1)} \]

where \( Y \) = property of steel and \( X_1, X_2, ..., X_n \) = process variables (i.e., in this case heat treatment / thermomechanical processing parameters).

Analytical expression for the above dependence is not known. We can present it in limits as a polynomial as:

\[ Y = b_0 + \sum_{i=1}^{n} b_i X_i + \sum_{i=1}^{n} \sum_{j=1}^{n} b_{ij} X_i X_j \]  
\[ \text{(1.2)} \]

Equation of the form (1.2) can be developed by mathematical analysis of the results obtained by factorial design of experiments. Use of such experiments permits investigation into the effects of number of different factors being varied simultaneously\(^{(2)}\). The treatment consists of all combinations that are possible from the different factors. Factorial design of experiments offer two advantages: (i) the least number of experiments need to be performed to get a relationship in
the form of an equation (thereby saving time, money and material), and (ii) the errors in the estimates are minimised due to confounding in the design matrix. The classical statistical methods of multiple correlation and regression on the other hand has the disadvantage that the errors due to personal, instrumental and conditional variation are not eliminated.

The response surface in terms of the main effects and interaction coefficients may be analysed to throw some light on the various physical and physico-chemical phenomenon occurring in the system. Further, help can be taken of the studies by Box and Wilson\(^{(3)}\), who studied the response surface in the neighbourhood of the maxima by the method of steepest ascent. This has been further developed by Box\(^{(4)}\) and others\(^{(5)}\). With these approaches it is possible to locate the maxima in the response surface.

If there are \('n'\) number of factors and each factor is varied at \('p'\) levels (limits), then the number of experiments required is \(p^n\). Thus a factorial design of the type \(2^2\) means there are 2 factors and each factor is varied at 2 levels. The insignificant terms in the equation obtained from the analysis of such experiments are eliminated by performing some standard statistical test ('\(t\)'-test). The adequacy of the equation is checked by variance ratio test ('\(F\)'-test)\(^{(6,7)}\). The validity of the equations is further checked by performing some random experiments and comparing the results with the values obtained from equations.

The details of statistical design of experiments, the methods of checking the adequacy of the equations obtained and optimization techniques are given in Chapter III.
1.5 Objectives of the Present Investigation

It is apparent that the properties of a high strength steel can be improved by heat treatment and thermomechanical processing. As stated earlier, development of mathematical model is necessary to predict the changes in microstructure and the properties due to heat treatment / thermomechanical processing. For this, one has to know the effects of the processing parameters on the microstructure and the mechanical properties.

The effect of the processing parameters (factors) on the properties (responses) of a steel can be known through experiments. The conventional way of experimentation is to vary one factor at a time, keeping the others at constant levels. However the results of these types of experiments do not accurately account for the effect on the response since the interactions occurring among these factors alter at different levels of the factors. Hence it is necessary to plan and conduct the experiments systematically and vary the factors simultaneously. This is possible through statistical design of experiments.

The objectives of the present work was thus to develop models to predict the changes in microstructure and mechanical properties of two HSLA steels (supplied by Naval Research Laboratory, U.S.A. and designated as GPT and GPP) due to heat treatment / thermomechanical processing and to predict the processing parameters that will produce the optimum/desired combination of properties. It was also thought to study the quantitative effect of process parameters (i.e., amount and temperature of deformation in the intercritical annealing zone) on the mechanical properties developed by thermomechanical processing of a dual phase steel with different starting microstructures.

The above objectives were achieved by carrying out the investigation as follows:
(i) Characterization of the two steels (GPT and GPP) in as-received condition,

(ii) (a) To study the quantitative effects of heat treatment parameters on the mechanical properties (hardness, tensile properties, low-temperature impact property) and microstructures (i.e., optical, SEM, TEM) of the heat treated steels,

(b) Optimization of mechanical properties,

(iii) (a) Design of TMCP schedule suitable for the GPP steel,

(b) Evaluation of mechanical properties and correlating them with grain sizes etc. for TMCP steel,

(c) Microstructural characterization of TMCP material (optical, TEM),

(d) Effects of tempering after TMCP on the microstructure and mechanical properties,

(e) Evaluation of contributions of various strengthening components to the Yield strength of a TMCP material,

(f) Effects of controlled cooling after controlled rolling.

(iv) Performing experiments with a dual phase steel (0.08-C, 1.00-Si, 1.21-Mn, 0.02-P, 0.012-S, 0.42-Cr, 0.41-Mo) to evaluate the effect of extent and temperature of deformation in the intercritical annealing range with different starting microstructures.

1.5.1 Characterization of As-Received Steels

The characterization of the two steels (GPT and GPP) in as-received condition were to be done with respect to (i) chemical composition, (ii) transformation temperatures, (iii) micro-structures (i.e., inclusion studies, grain
size analysis, optical, SEM, TEM) and (iv) mechanical properties (i.e., hardness, tensile properties, Charpy impact property at -50 °C), (v) fractography study (through SEM) of the fracture surfaces of broken tensile/Charpy specimens.

1.5.2 Characterization of Heat Treated Steels

Heat treatment studies were carried out for both the steels (GPT and GPP). The purpose of heat treatment experiments was to obtain a mathematical model to predict the mechanical properties (YS, toughness etc.) of the steels for changes in heat treatment process parameters. Further, heat treatment parameters for the optimum combination of properties as well as maximum toughness with a constraint on the strength property were aimed at. To meet the above requirements the following experiments were performed.

(i) To study the effects of austenitization temperature,
(ii) To study the effects of tempering temperatures (followed by air cooling / water quenching),
(iii) quantification of mechanical properties in the form of regression equations through (a) curve fitting technique (b) statistical design of experiments,
(iv) maximization / optimization of mechanical properties with respect to heat treatment process parameters using Steepest Ascent / Grid Search techniques respectively,
(v) microstructural characterization of the heat treated samples through optical, TEM studies,
(vi) examination of the fractured surfaces of the Charpy samples (broken at -50 °C).
1.5.3 Characterization of TMCP steel

Although a good deal of work has been reported on single-pass rolling, literature available on multi-pass rolling is comparatively less. The present work was designed to have multi-pass rolling keeping in view of the fact that industrial operation involves multi-pass rolling. Because of the similar compositions of GPP and GPT steels, it was thought to carry out TMCP operation with GPP steel only. The objectives (stated in Art. 1.5 (iii)) of TMCP operation were achieved through the following:

(i) design of rolling schedules suitable for the steel,
(ii) determination of the recrystallization stop temperature (since knowledge of this temperature is necessary to design the rolling schedules),
(iii) determination of flow stresses at different deformation temperatures,
(iv) evaluation of the dependence of flow stress on Zenner-Holloman parameter (i.e., the strain rate and deformation temperature),
(v) establishing relation between grain size with Zenner-Holloman parameter,
(vi) prediction of grain size after multi-pass rolling operation through modelling,
(vii) establishing regression equations for UTS and grain size correlating with TMCP parameters (i.e., temperature and amount of deformation),
(viii) constructing contour maps to help in selecting process variables for a desired property,
(ix) study the effects of tempering after TMCP on the mechanical properties and microstructure,
(x) microstructural characterization of TMCP material through optical microscopy and TEM studies,

(xi) determination of the contributions of the various strengthening components (i.e., solid solution strengthening, dislocation strengthening, precipitation strengthening and strengthening due to grain refinement) to the yield strength of a TMCP steel,

(xii) study of the role of deformation in the intercritical range with different starting microstructures. A dual phase steel was deformed through two different routes: (a) austenitising and cooling to the scheduled intercritical temperature and rolling to different extents at predetermined temperature, (b) heating the steel from the ferrite+pearlite condition to a predetermined temperature and deforming it to different extents at the scheduled intercritical temperature.

Details of experiments and discussions of results with conclusions are provided in respective chapters (please vide Contents). Modelling is never complete without verification of the validity of the models. Hence efforts were made to check the validity of the predicted results through statistical analysis as well as random experimentation.
References (Chapter I)