

## INTRODUCTION

### 1.1. Background :

Quenching and tempering behaviour of steels have been the focus of investigation since the turn of the century and fascinating references on the subject are available since very early times<sup>(1)</sup> to this date<sup>(2,3)</sup>. Apart from providing opportunities for making fundamental studies, the phase transformations associated with quenching and tempering have also attracted wide attention on account of their overwhelming influence on the resulting strength and fracture properties<sup>(4,5)</sup>.

It is now well established that the product of quenching in high carbon steels broadly consist of martensite, retained austenite and some undissolved carbides. As the austenitising temperature is raised above  $A_{Cm}$ , the carbides dissolve and amount of retained austenite is also affected<sup>(6-10)</sup>. It is also known that the extent of retained austenite is a very sensitive function of surface carbon and would be affected by a small decarburisation or carbon pick-up during austenitising. While alloying elements in small quantities are not known to affect the extent of retained austenite, the nature of carbide and its degree of dissolution during austenitisation may be affected.

The products of quenching tend to transform to stabler constituents when subjected to isothermal holding or continuous heating commonly known as tempering. In a

temperature range of 150<sup>o</sup>- 650<sup>o</sup>C, three stages have been identified and the general characteristics of processes occurring during the tempering have been more or less established<sup>(11)</sup>. In the first stage, a martensite of lower tetragonality along with a transition carbide form ; the second stage is associated with the transformation of retained austenite to lower bainite while the formation of cementite describes the third stage. The temperature and time-spans of these stages are known to overlap so that the above mentioned reactions can go on side by side.

While a general agreement exists on the broad features of the reactions during the various stages of tempering mentioned above, advent of modern techniques such as Mössbauer Spectroscopy, field-ion microscopy as well as more sophisticated interpretations of the data from established techniques such as electron-microscopy, X-ray diffractometry and neutron-diffraction have thrown more light in recent years on the processes occurring during the preliminary stages of tempering, preceding the already mentioned first stage. Thus, it is now reported that formation of clusters<sup>(11)</sup>, short-range ordering<sup>(12)</sup>/modulations, a different type of intermediate six-layer martensite<sup>(13)</sup> with tetra-hedral occupation of carbon-atoms<sup>(14-15)</sup> are possible in the freshly quenched high carbon-martensite, particularly at low temperatures. With tempering even at low temperatures these structures show changes towards the more commonly known structure in which the high-carbon martensite is b.c.t. with carbon-atoms essentially in the octahedral sites<sup>(16)</sup>.

The details of the transformation products in the three stages of tempering, more particularly the first stage has also seen the emergence of newer ideas. Thus - while earlier investigations<sup>(17-18)</sup> maintain it to be the h.c.p.  $\epsilon$  - carbide ( $\text{Fe}_{2.4}\text{C}$ ), some recent works<sup>(19-20)</sup> identify the carbides as  $\eta$  - carbide ( $\text{Fe}_2\text{C}$ ) with an orthorhombic structure. The process of conversion of high carbon martensite into the tempered products has also not been accepted with unanimity. While some researchers describe it as a 'two-phase decomposition' process with original high-carbon, high tetragonal martensite co-existing with a lower-carbon martensite<sup>(21-22)</sup>, others have shown it as a 'three-phase decomposition process'<sup>(2,23)</sup> particularly in the high-carbon steels where in, a martensite with intermediate carbon is believed to be occurring along side low carbon and high carbon martensites.

The '200'-martensite diffraction line along with the tetragonal doublets has been chosen in the past for studying the dipole distortion<sup>(24,25)</sup> in martensite owing to the fact that carbon atoms primarily occupy octahedral sites in the mid-points of C axes. The '211'-martensite lines displaying a doublet due to tetragonality has been used by investigators<sup>(2,26,27)</sup> for analysing the first stage of tempering, both in single crystals and in polycrystals. While some reports reveal that the '200'-doublet and '211'-doublet do not disappear with the same rate<sup>(27)</sup> other investigations report an agreement between the behaviour of the two lines<sup>(2)</sup> during tempering. The crystallography of the

precipitates forming in this stage has also been extensively studied<sup>(28-29)</sup>.

The decomposition of austenite into lower bainite and the subsequent appearance of cementite have been the subject of several investigations<sup>(30-32)</sup> and attempts have been made to estimate the corresponding activation energy.

Various aspects of the formation of cementite which constitutes the third stage, have been looked into by several workers and some consensus has been achieved with regard to its structure and morphology as well as crystallographic characteristics<sup>(32-34)</sup>. However, there are also some debatable aspects such as whether  $X\text{-Fe}_5\text{C}_2$  (Hägg carbide) appears or not as a precipitate in the early third stage of tempering (520-720K). Application of lattice and structure imaging technique<sup>(35)</sup> has shown that not only  $\theta\text{-Fe}_3\text{C}$  &  $X\text{-Fe}_5\text{C}_2$  but also higher order carbides, such as  $\theta_n\text{-Fe}_{2n+1}\text{C}_n$  ( $n \geq 3$ ) intergrow microsyntactically in the precipitated particles in the beginning of the third stage and gradually get transformed into  $\theta\text{-Fe}_3\text{C}$  in the temperature range 720-870 K.

Influence of several alloying elements on the tempering behaviour of steels has received wide attention and the literature<sup>(36-37)</sup> essentially shows that most alloying elements retard the processes of tempering.

## 1.2. Scope of the present investigation :

Preventing surface decarburisation in high carbon steels is quite an irritating problem, particularly with high austenitising temperatures. The first part of the present work was devoted therefore to the examination of the efficacy of some of the inexpensive coatings and atmospheres in this regard. Simultaneously, it was also thought worth while to evolve quick and accurate methods for checking up the surface carbon. The use of the tetragonal shift existing in the '200'-doublet was explored for this purpose.

Estimating the quantity of retained austenite in the quenched steel with the help of magnetic saturation measurements and the X-ray diffractometry, and examining the correspondence between the two techniques was undertaken.

The stabilisation of retained austenite is a fascinating phenomenon<sup>(38-41)</sup> of importance to researchers as well as the heat treaters. However, the tempering behaviour of a steel following a stabilising treatment has not received any attention so far and this was included for investigation in the present study. Similarly, the influence of the initial amount of retained austenite on the subsequent tempering behaviour has been incorporated in the present work.

The choice of high carbon steels was essentially prompted by the requirement to permit several levels of initial

austenite in the as-quenched condition for studying its influence. Along with a plain carbon steel, a ball-bearing variety with low Cr (1.6%) was considered appropriate to examine the possible effect of Cr on the tempering behaviour of a high carbon steel.

Since some ambiguity still exists regarding the rate of disappearance of the multiple components in the 200 and 211 martensite lines<sup>2,27</sup>, these two lines were chosen for conducting a number of kinetic studies with regard to the first stage of tempering. These lines were also thought suitable for examination of the possibility of co-existence of several martensites during the first stage of tempering.

It was further considered proper to study the austenite decomposition process by a relatively straight forward technique, such as the rate of lowering in the integrated intensity of the 311-austenite line. In order to examine the effect of stabilisation, a DTA method was utilised for direct comparison of stabilised and freshly quenched samples. It may be mentioned that DTA studies on the tempering of steels are rather scanty in literature. Keeping in view of the fact that magnetic saturation changes accompanying the transformation from paramagnetic austenite to ferromagnetic bainite are appreciable, this property was included for study too.

Further, coercive force changes being known as extremely structure sensitive, it has been thought important to monitor the variation in coercive force to examine the processes accompanying tempering and to compare the results with those obtained through other techniques.