PRECIPITATION BEHAVIOUR OF LOW CARBON MICROALLOYED STEEL

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Abstract. In this paper precipitation behaviour of low carbon micro alloyed steel is seen by various heat treatment cycles at different temperature ranges. Precipitation occurs during the ageing cycles and as we can conclude from harness graph and microstructural study that precipitation of alloys like NbC, SiC, CrC, NiC, VC, MoC, BC precipitates on higher temperatures, therefore hardness tends to increase at some range of high temperature ageing. After their precipitation occurs there got a saturation in the system and again hardness tends to fall. Although the hardness result of all the samples do not show very significant difference, but as a whole the test shows rise, saturation and fall in hardness values which is due to various precipitation temperatures of various alloying elements involved.

1. INTRODUCTION

At the end of the process of Quenching, second phase precipitation of microstructure occurs. At ambient temperature Precipitation with time is known natural ageing/maturing, whereas at elevated temperature precipitation is known as artificial ageing/maturing. After solution treatment hardness is comparatively less. The utmost strength and hardness develops when mixture is matured at a suitable temperature which varies between 120°C and 200°C. In few conditions the ageing temperature can be 300°C. Maturing duration may range from 4 hours to 24 hours. Unconstrained decay of supersaturately well build arrangement happens during maturing treatment. Aegering temperature and level of supersaturation assume an incredible function on the previous properties of the amalgam. More the level of immersion and the maturing temperature, more serious will be the maturing. Increased maturing temperature is embraced when steady stage is required along with dimension security. The cycle is additionally Termed as settling maturing. Other than mechanical properties, physico- substance properties are additionally affected by maturing. This occurs because of themetastable microstructure of the alloy that is framed during maturing of supersaturately strong arrangement got by arrangement treatment. Ascend in temperature varies the nuclear situations with relating variation in the powers related with interatomic bonds. Simultaneously circulation of second stage particles likewise changes. With variety in the assembling strategy of the segments, it shows variation in mechanical properties on higher temperatures after warmth heat treatment. It has been seen that the squeezed segments are a lot of better than the cast parts in this regard. Then again, the conduct of cast versus squeezed segments of silumin type compounds is the polar opposite. The distinction in conduct is because of the idea of encouraging stages at high temperatures.

1.1 A few stages are related with the cycle of precipitation solidifying like
(1) Formation of particles of the accelerating stage comprises of revision of iotas inside the gem cross section.
(2) Formation of progress structures as changed Guinier-Preston zones and middle stages. This may offer ascent to most extreme reinforcing in the compound.
(3) Development of stable stage from progress stages of particles haveing regular limits with the grain of the grid.
(4) development of the bigger grains to the detriment of the adjacent more modest grains. Because of this pressure help happens in the cross section as a rule at maturing elevated temperatures, which effects considerable increase in ductility and decrease in strength of alloy\textsuperscript{[10]}.

2. LITERATURE REVIEW

2.1. STEEL

The word steel is utilized for some extraordinary amalgams of iron. The combinations differ in manner they are formed and in the extents of the material alloyed to the iron, both. All prepares, in any case, contain modest quantities of carbon and manganese. As such, it very well may be termed that steel is a glasslike amalgam of I-C and a few different components, which solidifies over its basic temperature. Like expressed above, there do exist a few kinds of prepares however commonly these order in two classifications are plain carbon and alloyed steels\textsuperscript{[2]}.

2.1.1. PLAIN CARBON STEELS

Plain carbon steel finds various applications and around 90\% of absolute steel creation falls into this class. The purposes behind quite a wide pertinence are its minimal effort combined with sensibly great usefulness, castability and machinability. The properties of carbon prepares can be changed fundamentally by basic mechanical, warm and surface medicines. These prepares, in different structures, are broadly utilized for general designing purposes. The different applications incorporate wires and wire items, rounded items, sheets and strips, railroad rails, projected and fashioned items, cutting devices, and so on. All steels contain silicon and manganese. Hence, it becomes necessary to define limits for silicon and manganese in order to differentiate between carbon steels and alloy steels. The American Iron and Steel Institute (AISI defines carbon steel as that steel for which no minimum content is specified or required for aluminium, chromium, cobalt, niobium, molybdenum, nickel, titanium, tungsten, vanadium, or zirconium, or any other element added to obtain desired alloying effect; when the specified minimum for copper does not exceed 0.40 per cent; or when the maximum content specified for any of the following elements does not exceed the percentage noted against them;

Manganese 1.65 percent
Silicon 0.60 percent
Copper 0.60 percent

In simple words, a steel containing less than per cent manganese and 0.60 per cent silicon is referred to as carbon steel\textsuperscript{[3]}.

2.1.2. CLASSIFICATION CARBON STEELS

Plain carbon steels can be divided into various classes based on carbon content, application and steel manufacturing method. Arrangement on the bases of carbon content is famous and habitually utilized. For the most part, with an expansion in the carbon content from 0.01 to 1.5\% in the combination, its quality and hardness increments yet such an increment past 1.5\% causes apparent decrease in the flexibility and capliability of the steel. As indicated by carbon content, carbon prepares might be delegated:

a. Low C steels (carbon less than 0.30\%)
b. Medium C steels (carbon between 0.30 to 0.60\%)c. High C steels (carbon greater than 0.60\%)
d. Hypoeutectoid steels (carbon less than 0.80\%),e. Eutectoid steels (carbon = 0.80\%)f. Hypereutectoid steels (carbon greater than 0.80\%)
Low C steel or mellow steel, constitutes carbon up to 0.30% reacts to moderate heat treatment as progress in flexibility is considered however has nul impact in regard of its properties. Medium C steels having carbon constitute going from 0.30 to 0.60% improves machin ability by heat treatment. It should likewise be noticed that the steel is particularly versatile for producing and where hardness is alluring.

High C prepared, is steel consisting carbon in the scope of 0.60 to 2.0% and particularly classed as high carbon steel. In completely heat treat condition, it is extremely hard and it will withstand higher wear, shear and will in this way be exposed to minimum distortion.

In view of uses, steel might be named auxiliary steel, constructional steel, apparatus steel, and so on. These prepares can additionally be partitioned based on explicit applications, for example rail steel, spring steel, heater steel, sheet steel, cold heading steel, and so forth.

Steel fabricating technique greatly affects the properties and nature of steel. Subsequently it has been a training to specify the steel fabricating technique up to mid-eighties; electrically liquefied prepares were utilized to be indicated by setting letter “E” before the AISI/SAE assignment of prepares. Characterization dependent on steel fabricating technique isn’t famous these days because of progressions in steel making innovation. Earlier, in view of steel making strategies, prepares were known as corrosive bessemer prepared steel, open hearth prepared steel, spring prepared steel, and L.D. prepared steel etc.

2.2. ALLOY STEELS

The restrictions of plain carbon prepares can be limited by the expansion of at least one components to plain carbon prepares. Carbon prepares accordingly alloyed with components are alluded to as amalgam prepares. Hence composite steel is the one that contains determined amount(s) of alloying element(s) and additionally more than 1.65 percent manganese, 0.60 percent copper as well as percent silicon.

The properties of composite prepares rely upon both carbon and alloying components. Nature and amount(s) of alloying element(s) direct the designing conduct of amalgam prepares. Alloying components not just limit/wipe out the restrictions of plain carbon prepares yet in addition confer/improve explicit attributes, for example, obstruction against consumption and oxidation, and attractive and electrical properties.

Cobalt, copper, titanium, tungsten, lead, manganese, molybdenum, nickel, silicon, Aluminium, boron, chromium and vanadium are few common mixing elements added to Steels.

2.2.1. CLASSIFICATION OF ALLOY STEELS

Alloy steels can be classified in to various classes depending on various parameters. Some popular bases for alloy steels classification are:

a. Amount of alloying element(s).
b. Principal alloying element(s).
c. Application of steels.
d. Microstructure of steels.

Based on amount of alloying element(s) available in steels, alloy steel also referred as low alloy steel, medium alloy steels and high alloy steels. In general, a low alloy steel contains up to 5% total alloy content while total alloy content of a high alloy exceeds 10%. For a Steels can be grouped as pearlitic steels, ferritic steels, martensitic steels, bainitic steels, and austenitic steels on the bases of microstructure of steels.
Table 1: Table summarizing the various effects of different alloying elements in the steel.

| Alloying/mixing element | Cr  | Co  | Mn  | Mo  | Ni  | Ti  | W  | V  |
|-------------------------|-----|-----|-----|-----|-----|-----|----|----|
| Hardenability           | +2  | +2  | +2  | +1  | +2  | +2  | +3 |    |
| High temperature strength| +1  | +1  | +2  | +1  | +2  | +2  |    |    |
| Ductility and toughness |     | +1  |     |     |     |     |    |    |
| Abrasion resistance     | +1  | +1  | +1  | +2  | +1  |     |    |    |
| Fine austenite grain size| +1  | +1  | +1  |     | +3  |     |    |    |
| Corrosion resistance    |     |     | +1  |     |     | +1  |    |    |

2.2.2. HOW DO MIXING/ALLOYING HELPS IN ACHIEVING OF MARKABLE IMPROVEMENT IN PROPERTIES OF STEEL?

Truth be told, the properties of compounds are very reliant on the connection between synthetic creation, preparing and their microstructure. For example, at whatever point a component is added to an unadulterated metal, the last modifies the size of the cross section structure of the metal and relying upon the composite framed, it can likewise change its grid type. In some cases metals do respond together to shape intermetallic mixes with exceptionally complex cross section structure. Such mixes soften at a fixed temperature and have a lower conductivity and pliability however more prominent quality and hardness than a combination of face focused body, focused or hexagonal grid structure. Hence, combinations increment quality and hardness of metal by changing its structure. Besides, as expressed above, alloying empowers the development of fine grain size since it favors the ability of the metal to be hardened by quenching in oil or air rather than in water. Indeed, oil is a cooling agent offering slow cooling rate and thus the grain form more regularly with time and hence they are finer\(^2,3,5\).

2.2.3. COMPOSITION VARIANCE OF FEW ELEMENT AND THEIR EFFECT ON THE FINAL ALLOY

Likewise alloying elements are used in steel so as to improve its properties and this is in the scope of rendering the steel appropriate for different uses. Alloying is not only a matter of adding elements to steel, the quantity of alloying elements added is of prior importance so as to achieve the required hardness, toughness or machinability of steel.

Table 2: shows the different composition variance used in alloying element with their effect on the final steel\(^2,3,5\).

| Alloying element | Range/variance | Effect on properties |
|-----------------|----------------|---------------------|
| Carbon(C)       | 0% to 0.25%    | Increases heat treatment and ductility.|
|                 | 0.25% to 0.70% | Increases machinability.|
|                 | 0.70% to 1.50% | Improvement in strength and hardness.|
|                 | More than 1.50%| Decrease in ductility and malleability.|
| Silicon         | Initial to 0.30%| Improves good casting fluidity.|
|                 | 0.30% to 1%    | Improves heat resistance.|

4
| Element  | Amount       | Effect                                                                 |
|----------|--------------|------------------------------------------------------------------------|
| Copper   | 0.20 to 0.50% | Increases steel resistance to atmospheric corrosion. Unwanted to steel as it affects surface finish. |
|         | High amount  |                                                                        |
| Chromium | 0 to 5%      | Quenching speed reduction, increased toughness, and wear resistance    |
|         | Upto 14%     | Resistance to corrosion improves, critical temperature.                |
| Manganese| 1.65 to 2.10%| Increases electrical resistance and magnetic property of steel and reduces its coefficient of expansion. Increases hardness and toughness. |
|         | 10% to 14%   |                                                                        |
| Tungsten | 3 to 6%      | Increases cutting hardness and abrasion resistance.                    |
| Vanadium | 0 to 0.05%   | Hardenability is increased.                                            |
|         | Less than 0.25% | Induces resistance to softening at high temperatures.                  |
|         | 1%            | Retain hardness at high temperature.                                   |
| Nickel   | 0 to 5%      | Provides refined grain structure and causes hardening abilities.       |
|         | 8 to 12%     | Resistance to low temperature impact.                                  |
|         | 15 to 25%    | High magnetic properties are imparted to steel.                       |
|         | 25 to 35%    | Corrosion resistance is increased.                                     |
|         | 36%          | Invar is obtained, which has a low coefficient of temperature.         |
| Molybdenum | 0.15 to 0.25% | Uses in combination with chromium, it improves ultimate strength of steel without affecting ductility or workability. |
| Lead    | 0.15 to 0.35% | Improves machinability.                                               |
| Boron   | 0.0005 to 0.03% | Improves hardenability of steel.                                     |
2.2.4. **LOW CARBON MICROALLOYED STEELS**

It holds an important category of steels estimated to be around 12% of total world steel production. These steels contain 0.07 to 0.12% carbon, up to 2% manganese and small additions of niobium, vanadium and titanium (usually max. 0.10% and total microalloying elements generally less than 0.15%) in various alloying. These steels stand in terms of performance and cost, between carbon steel and low alloy steel. These are used in major steel market sector in many parts of the world and their development has played an important role in the expansion of certain key industries such as oil and gas extraction, construction, and transportation[6]. The first microalloying element to be widely used was vanadium added to C Mn steels in the USA as reported by Bullens in 1916. Indeed early in the 20th century Hemp Ford made major utilisation of vanadium steels for the construction of its Model T, the first mass produced motor car. Small titanium additions can also improve the strength of steel and this was first exploited in Germany in 1921. Micro titanium and micro vanadium additions began to be used in China & India in the 1950s and 1960s for large diameter oil and gas pipelines. The requirement was high strength as compared to mild carbon steel, combined with improved toughness and good weldability. The material is preferably produced by a thermomechanical (hot-cold) rolling process, which maximizes grain refinement as a basis for improved mechanical properties. Machinability is improved than QuandT steels because of their more uniform hardness and their ferrite-pearlite microstructure. Because microalloyed steels are not quenched and tempered they are not susceptible to quench cracking, nor do they need to be straightened or stress relieved[7]. Molybdenum & vanadium has an important role in the initial development. The addition of 0.1-0.2% molybdenum produces a fine grain structure of acicular ferrite and substantially enhances the precipitation hardening effects achieved with the other alloying elements. Consequently, an estimated 2 million tons of Mo containing micro alloy steels for pipelines have been produced worldwide during the 1970s. Low carbon microalloyed steels, are designed to provide better mechanical properties and/or greater resistance to atmospheric corrosion than conventional carbon steels. They are not considered to be alloy steels in the normal sense because they are designed to meet specific mechanical properties rather than a chemical composition[8].

**Applications:**
- In Rail road construction
- In Off-shore/Platforms formation
- In Bridge element construction
- In Suspension part production
- In preparing building Structural components
- In Heavy Equipment produce
- In Tubular Components production
- In Vehicles/Transportation

| Table 3. Chemical composition of low carbon micro alloy steel sample (PB53306) |
|-----------------|---------|---------|
| Alloying element | Maximum | Sample composition |
| C                | 0.090   | 0.061   |
| Mn               | 1.70    | 1.59    |
| S                | 0.008   | 0.002   |
| P                | 0.018   | 0.007   |
| Si               | 0.45    | 0.193   |
| Cr               | 0.350   | 0.215   |
| Ni               | 0.50    | 0.013   |
3. GRAIN SIZE REDUCTION IN STEEL

It is known that mechanical properties of metals depend on their grain size. The Hall-Petch ($\sigma_0 = \sigma_1 + K_D^{-1/2}$) formula is an example of the reliance in which the yield stress of polycrystalline materials is invert to the grain size of material. Where $\sigma_0$ is the flow stress, $\sigma_1$ the fractional stress resisting the motion of a dislocation, $K_D$ the extent to which dislocations are collided at grain barriers, and $D$ is the grain average dia. This suggests, the finer the grain size is the greater the value will be of flow stress. Grain size reduction is the only commercially available conventional technique which increases ductility and strength simultaneously [8].

Figure 3. shows variation in the yield strength of LCS (low C steel) as a function of its grain size. It can be seen that reduction of ferrite grain size varies from 10μm - 0.5μm, increases the yield strength by 400MPa without any addition of alloying elements. The increase in strength can be seen due to the significant numbers increase of grain boundaries/unit volume.

| Element | Cu  | Ti  | V   | Nb  | Mo  | Al  | N   | Ca  | B   | Nb+Ti | Al/N 2:1 | Sn  | As  | Sb  | Pb  | Bi  | CE(HW) | CE(PCM) |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-------|----------|-----|-----|-----|-----|-----|--------|---------|
|         | .200| 0.025| 0.070| .100| 0.25| 0.050| 0.0120| 0.005| 0.0005| .150 | 12.37 | 0.015 | 0.020| 0.010| .010| 0.010| -     | 0.210 |

Fig 3. yield & tensile strength with different grain size of steels
Grain size shows effects on transition temperature. Grain coarsening effects the transition temperature and makes steel more suitable to fail by brittle fracture. Grain size effect on the transition temperature is more affective in the of low carbon steels. It is not easy to derive a mathematical relation in grain size and creep strength, because variation in grain size for creep study or high temp strength, is always followed by other changes. Coarser grain steel has better creep/high temperature strength above the equi cohesive temperature. Below equicohesive temperature, finer grain steels shows better high temperature strength. It has been observed that cast steels have incresed high temperature strength over forged steels. Coarse grain size of cast steel is believed to be responsible for the phenomenon. Similar reason is given for better high temperature strength of silicon deoxidized steels as compared to aluminum deoxidized steels. Fatigue strength does not exhibit any basic relationship with grain size. However, coarse grained steels have lowe fatigue strength as compared to fine grained steels. This is true provided the temperature of the test piece is not so high that creep is a dominating marvel. Coarse grained steel has preferred hardenability over fine grained steel. The explanation behind this is that coarse grained steel has less grain limits. Grain limits are where the pace of dissemination is high. Thusly, arrangement of pearlite, which is a dispersion controlled cycle, begins at grain limits. With more modest grain limit region in coarse grained prepares, special development of martensite from austenite happens on cooling. It will additionally be applicable to take note of that pearlite arrangement continues by nucleation and development instrument. Austenitic grain limits go about as nucleation locales for pearlite arrangement.

Fine grain steels have poor machin ability than coarse grain steels. Coarse grained steel has reduced toughness and responsible for providing small discontinuous chips during machining. In low C Mn-Si steels the DBTT, the temperature at which 50 % of the fracture area is covered by brittle area, can be lowered with grain size reduction. It can be said that the grain size reduction will increase fatigue and wear properties of low carbon steels. Grain refinement increases s toughness as well as improves strength of the materials and provides scope for research. If grain refinement is done to the nano meter level, the nanocrystalline metals have improved mechanical properties in comparison to their coarse-grained metals, results in various applications[1,9].

4. EXPERIMENTAL PROCEDURE

Various processes has been carried out in the presented project in order to know about the precipitation behaviour in low carbon micro alloyed steel whose chemical composition is given in table 3. Some steps during process are likewise:

a. Samples were cut with the help of circular saw and hand saw to avoid over heating during cutting action.

b. Afterwards samples were heated to a austenitising temperature of 11500C in a muffle furnace for 1hour holding time.

c. Then samples were quenched in a ice brine solution.

d. Hardness of the as quenched sample were measured by means of Vicker's hardness test.

e. Afterwards samples were aged at various temperatures like 100,200, 300,400,500,600,625,650,675,700oC for holding time of 1hour each.

f. Then hardness of the samples were measured by means of Vicker’s hardness test and a graph were plotted between Vicker's hardness number (VH) and temperature according to the results to know the effect of precipitation on hardness of sample.

g. Thereafter microstructure study were carried out on the samples having most significant difference in hardness numbers by means of optical microscopy.

5. RESULTS AND DISCUSSION

As we can take two phases of result according to our experiment:

5.1. ACCORDING TO THE HARDNESS NUMBER
Table 4. Shows various differently treated samples at varying temperature and their hardness by Vicker’s hardness testing machine.

| S.No. | Sample type                        | Ageing Temperature (°C) | VHN |
|-------|------------------------------------|-------------------------|-----|
| 1     | Before processing                  | NIL                     | 205 |
| 2     | Aged Samples for 1 hour each       | 100                     | 218 |
|       |                                    | 200                     | 218 |
|       |                                    | 300                     | 219 |
|       |                                    | 400                     | 225 |
|       |                                    | 500                     | 235 |
|       |                                    | 600                     | 252 |
|       |                                    | 625                     | 220 |
|       |                                    | 650                     | 238 |
|       |                                    | 675                     | 210 |
|       |                                    | 700                     | 229 |
| 3     | As quench samples (1150°C/water quenched) | NIL                  | 215 |

We take the values having significant difference during ageing cycle like VH 218, 235, 252 and 238. We took the values and plotted a graph between hardness number and temperature of ageing cycles which is further shown in fig 4. Also, we can see a difference in hardness after quenching.

Fig 4. Graph between Vicker’s hardness number and ageing temperature

As the graph shows there, we got an increase in hardness to an ageing temperature limit and falls back again. This is due to high temperature precipitation of some alloys like NbC.

5.2. ACCORDING TO MICROSTRUCTURAL STUDY

The microstructures of selected samples are:

Fig 5. microstructure of aged sample at 200°C For 1 hour at 1000x magnification
As we can see from the images above that there are precipitated particles in the microstructure and also the precipitation of high temperature precipitating alloy like NbC in the microstructure. Hardness increases up to the ageing temperature of 600°C. There are 10% Pearlite, a little amount of Upper Bainite, Ferrite and some corrosion product due to straining at the grain boundaries in the microstructure. Then after the temperature of 600°C, precipitation of high temperature precipitants occurs and there got a saturation and hardness tends to decrease due to coarsening of precipitates.
6. CONCLUSION

The study shows that precipitation occurs during the ageing cycles and as we can conclude from harness graph and microstructural study that precipitation of alloys like NbC, SiC, CrC, NiC, VC, MoC, BC precipitates on higher temperatures, therefore hardness tends to increase at some range of high temperature ageing. After their precipitation occurs there got a saturation in the system and again hardness tends to fall. Although the hardness result of all the samples do not show very significant difference, but as a whole the test shows rise, saturation and fall in hardness values which is due to various precipitation temperatures of various alloying elements involved.

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