RESULTS AND DISCUSSION

4.1 OPTICAL METALLOGRAPHY

Figures 4.1 - 4.17 shows the optical micrographs of sheet steel after various stages of thermal processing. The as received steel showed the predominantly ferrite and retained austenite with a few of coarse carbide precipitate particles along the grain boundaries (Figs.4.1). Fig. 4.1 shows the existence of a few coarse fine precipitates within the grains. Solution treatment at 1000°C followed by oil quenching results in the fragmentation of grain boundaries and coarsening of precipitates particles (Figs. 4.2).

4.1.1 Effect of Aging at 400°C

Figure 4.3 - 4.10 show the optical micrographs after various stages of Aging at 400°C. It is observed that Aging at 400°C results in coarsening and dissolution of precipitate particles together with segregation along the grain boundaries. Aging for 1 min results in coarsening of grains and precipitate particles (Figs. 4.3). Subsequent Aging to 15 min reveals the grain refinement associated with few precipitate particles along the grain boundaries (Figs. 4.4).

However, Aging for 60 min results in the formation of fine carbide precipitate particles in the ferrite matrix together with the fragmentation of grains (Figs. 4.5). It is also observed that Aging for 60 min causes the formation of acicular polygonal ferrite. Subsequent Aging leads to the coarsening of grains together with segregation of carbide precipitate particles along the grain boundaries (Figs. 4.6 - 4.8). Further Aging to 20 hr causes the coarsening and segregation of carbide precipitate particles along the grain boundaries (Figs. 4.9). A few fine precipitates are also observed within the grains. Moreover, prolonged Aging leads to the formation of fine ferrite grains free from carbide particles after 50 hr (Fig.4.10).
Figure 4.1: Optical micrograph of sheet steel in As-received Condition.
Figure 4.2: Optical micrograph of sheet steel after solution treatment at 1000°C.
Figure 4.3: Optical micrographs of sheet steel after 1 min of aging at 400°C.
Figure 4.4: Optical micrographs of sheet steel after 15 min of aging at 400°C.
Figure 4.5: Optical micrographs of sheet steel after 1 hr of aging at 400°C.
Figure 4.6: Optical micrographs of sheet steel after 2 hr of aging at 400°C.
Figure 4.7: Optical micrographs of sheet steel after 5 hr of aging at 400°C.
Figure 4.8: Optical micrographs of sheet steel after 10 hr of aging at 400°C.
Figure 4.9: Optical micrographs of sheet steel after 20 hr of aging at 400°C.
Figure 4.10: Optical micrographs of sheet steel after 50 hr of aging at 400°C.
4.1.2 Effect of Aging at 500°C

Figures 4.11 - 4.17 shows the optical micrographs of the sheet steel in various stages of Aging at 500°C followed by water quenching. In general, aging at 500°C results in the formation of fine/coarse carbide precipitate particles. The particles in general are observed within the ferrite matrix and along the grain boundaries. Aging for 1 min results in the formation of coarse grains with a very few fine precipitate (Figs. 4.11). Subsequent Aging to 15 min results in the fragmentation of grains with enhanced density of precipitates within the matrix as well along the grain boundaries (Figs. 4.12).

Further aging to 1hr results in the fragmentation of ferrite with uniform distribution of fine precipitate particles in the matrix (Figs. 4.13). However, subsequent aging to 2 hr causes the segregation of precipitate particles along the boundaries leaving only a very few precipitate particles in the matrix (Figs. 4.14). Figs. 4.15 reveals the refinement with only a few precipitate particles in the ferrite matrix. Subsequent aging to 10 hr results in the coursing of grain and carbide precipitate particles (Figs. 4.15).

However, as the aging proceeds alternately dissolution of pre-existing precipitate particles and formation of new fine/coarse precipitate particles take place which is also associated with the defragmentation of grains (Figs. 4.16 - 4.17).
Figure 4.11: Optical micrographs of sheet steel after 1 min of aging at 500°C.
Figure 4.12. Optical micrographs of sheet steel after 15 min of aging at 500°C.
Figure 4.13: Optical micrographs of sheet steel after 1 hr of aging at 500°C.
Figure 4.14: Optical micrographs of sheet steel after 2 hr of aging at 500°C.
Figure 4.15: Optical micrographs of sheet steel after 10 hr of aging at 500°C.
Figure 4.16: Optical micrographs of sheet steel after 50 hr of aging at 500°C.
Figure 4.17: Optical micrographs of sheet steel after 100 hr of aging at 500°C.
4.2 HARDNESS

The as received steel showed a hardness value of 137 HV10. Solution treatment at 1000°C for 3hr followed by oil quenching significantly increases the hardness to 154 HV10. Figure 4.18 show the variation of hardness with aging time.

In general, the variation of hardness with aging at 400°C and 500°C follows a similar trend. This is evident by the multiple peaks in the aging curve. These variations in hardness are attributed to the formation and segregation of coarse fine carbide precipitate particles. As evident, from the figure, the hardness first increases attained a peak value after 15min at 400°C then decreases on subsequent aging to 30 min. However further, aging again increases the hardness after 60min. It is observed that aging beyond 60min again decreases the hardness value significantly. Aging at 500°C follows a similar trend. However the process of aging is slow at 500°C as compared to that at 400°C. This is evident, from the Fig 4.18 that first peak at 500°C is attained after 60min, which corresponds to the second peak at 400°C. The higher hardness values in peak aged conditions are attributed to the formation of fine precipitate particles within the grains together with grain refinement. However, the significant drop in hardness values are associated with the dissolution, coarsening and segregation of precipitate particles within and along the grain boundaries.
Figure 4.18. Variation of hardness (HV) as a function of aging time.
4.3 DISCUSSION

Age hardening in metals and alloys has many practical applications. Precipitation hardening, also called age hardening, is a heat treatment technique used to increase the yield strength of malleable materials. Precipitation hardening relies on changes in solid solubility with temperature to produce fine particles of an impurity phase, which impede the movement of dislocations, or defects in a crystal's lattice. Since dislocations are often the dominant carriers of plasticity, this serves to harden the material. The impurities play the same role as the particle substances in particle-reinforced composite materials. Just as the formation of ice in air can produce clouds, snow, or hail, depending upon the thermal history of a given portion of the atmosphere, precipitation in solids can produce many different sizes of particles, which have radically different properties. Unlike ordinary tempering, alloys must be kept at elevated temperature for hours to allow precipitation to take place.

Two different heat treatments involving precipitates can alter the strength of a material: solution heat treating and precipitation heat treating. Solid solution strengthening involves formation of a single-phase solid solution via quenching. Precipitation heat treating involves the addition of impurity particles to increase a material's strength (Callister, 2nd ed. Wiley & Sons. pp. 252).

This technique exploits the phenomenon of supersaturation, and involves careful balancing of the driving force for precipitation and the thermal activation energy available for both desirable and undesirable processes. Nucleation occurs at a relatively high temperature (often just below the solubility limit) so that the kinetic barrier of surface energy can be more easily overcome and the maximum
number of precipitate particles can form. These particles are then allowed to grow at lower temperature in a process called aging. This is carried out under conditions of low solubility so that thermodynamics drive a greater total volume of precipitate formation.

Diffusion's exponential dependence upon temperature makes precipitation strengthening, like all heat treatments, a fairly delicate process. Too little diffusion (under aging), and the particles will be too small to impede dislocations effectively; too much (over aging), and they will be too large and dispersed to interact with the majority of dislocations.

Precipitation strengthening is possible if the line of solid solubility slopes strongly toward the center of a phase diagram. While a large volume of precipitate particles is desirable, a small enough amount of the alloying element should be added that it remains easily soluble at some reasonable annealing temperature.

Many alloy systems allow the aging temperature to be adjusted. For instance, some aluminium alloys used to make rivets for aircraft construction are kept in dry ice from their initial heat treatment until they are installed in the structure. After this type of rivet is deformed into its final shape, aging occurs at room temperature and increases its strength, locking the structure together. Higher aging temperatures would risk over-aging other parts of the structure, and require expensive post-assembly heat treatment. Too high of an aging temperature promotes the precipitate to grow too readily.

The primary species of precipitation strengthening are second phase particles. These particles impede the movement of dislocations throughout the
lattice. You can determine whether or not second phase particles will precipitate into solution from the solidus line on the phase diagram for the particles. Physically, this strengthening effect can be attributed both to size and modulus effects, and to interfacial or surface energy.

The presence of second phase particles often causes lattice distortions. These lattice distortions result when the precipitate particles differ in size and crystallographic structure from the host atoms. Smaller precipitate particles in a host lattice leads to a tensile stress, whereas larger precipitate particles leads to a compressive stress. Dislocation defects also create a stress field. Above the dislocation there is a compressive stress and below there is a tensile stress. Consequently, there is negative interaction energy between a dislocation and a precipitate that each respectively causes a compressive and a tensile stress or vice versa. In other words, the dislocation will be attracted to the precipitate. In addition, there is positive interaction energy between a dislocation and a precipitate that have the same type of stress field. This means that the dislocation will be repulsed by the precipitate.

Precipitate particles also serve by locally changing the stiffness of a material. Dislocations are repulsed by regions of higher stiffness. Conversely, if the precipitate causes the material to be locally more compliant, then the dislocation will be attracted to that region.

Furthermore, a dislocation may cut through a precipitate particle. This interaction causes an increase in the surface area of the particle. The area created is
\( A = 2\pi r b \)

where, \( r \) is the radius of the particle and \( b \) is the magnitude of the burgers vector. The resulting increase in surface energy is

\[ E = 2\pi r b \gamma_s \]

where \( \gamma_s \) is the surface energy.

The dislocation can also bow around a precipitate particle. There are two equations to describe the two mechanisms for precipitation hardening:

Dislocations cutting through particles:

\[ \tau = \frac{\pi r \gamma}{b L} \]

where \( \tau \) is material strength, \( r \) is the second phase particle radius, \( \gamma \) is the surface energy, \( b \) is the magnitude of the Burgers vector, and \( L \) is the spacing between pinning points. This governing equation shows that the strength is proportional to \( r \), the radius of the precipitate particles. This means that it is easier for dislocations to cut through a material with smaller second phase particles (small \( r \)). As the size of the second phase particles increases, the particles impede dislocation movement and it becomes increasingly difficult for the particles to cut through the material. In other words, the strength of a material increases with increasing \( r \).

Dislocations bowing around particle:

\[ \tau = \frac{G b}{L - 2r} \]

where \( \tau \) is the material strength, \( G \) is the shear modulus, \( b \) is the magnitude of the Burgers vector, \( L \) is the distance between pinning points, and \( r \) is the second
phase particle radius. This governing equation shows that for dislocation bowing
the strength is inversely proportional to the second phase particle radius \( r \).
Dislocation bowing, also called Orowan strengthening, is more likely to occur
when there are large particles present in the material
(http://aluminium.matter.org.uk).

These governing equations show that the precipitation hardening
mechanism depends on the size of the precipitate particles. At small \( r \), cutting will
dominate, while at large \( r \), bowing will dominate. Looking at the plot of both
equations, it is clear that there is a critical radius at which max strengthening
occurs. This critical radius is typically 5-30 nm.

The precipitation behavior and texture formation during hot rolling, cold
rolling and annealing (at 700°C and 800°C) in a Ti-containing interstitial free high
strength (IFHS) steel (Ghosh et al. 2008) revealed that annealing at 700 and
800°C results in a completely different type of precipitation behavior. Annealing at
700°C produces copious FeTiP type precipitates, while after annealing at 800°C, Ti₄C₂S₂ precipitates form. Due to the formation of Ti₄C₂S₂, C is easily taken out of the solid solution during recrystallization annealing and this results in better {111} texture formation and thus better formability in the 800°C annealed material. Formation of FeTiP in the 700°C annealed material leaves behind less Ti for TiC precipitation during recrystallization annealing. This results in the matrix becoming highly free, leading to poorer {111} texture formation as well as poorer adhesion, the fine FeTiP precipitates sitting on the grain boundaries affect the sharpening of {111} recrystallization texture.

**REBCES**

http://aluminium.matter.org.uk

Ghosh et al. 2008
