EFFECTS OF AGEING TREATMENT ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Cu – ADDED Ti, B MICROALLOYED STEELS

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ABSTRACT

The present investigation concerns the influence of Cu on the ageing behavior of the Cu - added Ti, B microalloyed steels subjected to air cooling and water quenching after hot rolling. The microstructural evolution and mechanical properties achieved in the cases of Cu - added and Cu - less steels has unveiled the favorable role of Cu in forming the ferrite - martensite dual phase structure after air cooling and bainitic structure after water quenching of the hot rolled steel. The Cu - added samples after hot rolling followed by air cooling and water quenching are subjected to the isochronal ageing at appropriate temperatures with and without 15% cold deformation prior to the ageing. After carrying out ageing treatment of the hot rolled samples without any cold deformation, nominal improvement in the hardness value has been recorded at the peak ageing temperatures. Air cooled and water quenched samples after 15% cold deformation have yielded significant improvement in hardness by subsequent ageing treatment. It has also been demonstrated that a suitable amount of cold deformation prior to the ageing treatment aids in lowering the peak ageing temperature in comparison to the samples aged without any prior cold deformation.

1. INTRODUCTION

During the last two decades an extensive effort has been launched in the direction of development of high strength steels capable of providing adequate ductility, formability and fracture toughness. The demand for such steels was realized in automobile, defense and naval sectors. Dual phase (DP) steel has been slotted as one of the potential candidates in this group, which comprise hard martensite particles dispersed in the soft ferrite matrix, where the matrix ensures high formability and the hard dispersed phase provides the strengthening effect. In spite of the fact that DP steels adequately fulfill the requirements for the thin gauged application, further improvement of the mechanical as well as corrosion property of the DP steels is eagerly awaited for its application in defense, naval and other heavy duty sectors. In this regard, it may be mentioned that the major concern regarding the broad application of thin gauged high strength steels is the manufacturing cost contributed by the necessity of alloying elements and installations for adopting the thermomechanical processing routes. Other limitations of significance are the formability and weldability, which restrict the wider use of high strength steel sheets.

A review of the current status of the conventional high strength steels including the DP steel has indicated that production of such steels by adopting further simplified processing route without causing any significant detriment of mechanical properties and yield, is essentially warranted. A judicious approach in this direction may be the improvement of strength in the case of DP steel in one hand, and improvement of the formability of high strength bainitic steels on the other. This may be achieved by appropriate microstructural engineering.
incorporating affordable alloying addition without the necessity of complicated thermo mechanical processing.

In the present investigation, a Ti, B microalloyed steel has been alloyed with 1.5 wt % Cu to utilize the individual effect of Ti, B and Cu, and synergistic effect of the combinations of Ti, B and Cu, B with respect to enhancement of the hardenability of austenite and thereby providing the opportunity to obtain DP or bainitic microstructure under the affordable thermomechanical processing schedule. Moreover, an attempt has been made to study the possibility of hardening the hot rolled steel by subjecting the same to various ageing treatments with and without suitable amount of prior cold rolling. It may be noted here that in the present phase of investigation particular emphasis has been given to the possibility of the improvement of mechanical properties by adopting affordable alloying and process schedule while detailed characterization of the microstructural constituents and identification of the concerned mechanisms remain limited within the scope of scanning electron microscopy.

2. EXPERIMENTAL DETAILS

The predetermined charge blends were induction melted under controlled condition suitable for the sequential additions of the alloying as well as microalloying elements followed by homogenization at 1200°C and subsequent forging for complete elimination of the cast structure. The chemical analysis of the samples collected from the forged ingot was carried out in a Spectroscopic (Spectrolab ccd) analyser and the results are presented in Table 1. Subsequently, the samples were homogenised at 1200°C and were subjected to rolling with a finish rolling temperature (FRT) of 750°C, followed by air cooling (AC) for one set of samples and water quenching (WQ) for the other. Some samples were hot rolled down to a thickness of 6 mm. A few samples with FRT of 750°C were rolled down to 7 mm, which were then subjected with a 15% cold working (CW) to reduce the thickness down to 6 mm. The effects of variation of the hot rolling parameters on the microstructural evolutions have been examined by subjecting the samples to optical and scanning electron microscopic (JEOL, JSM-5800 scanning microscope) investigations. The microstructures have been studied by using different magnifications and the most appropriate ones revealing the desired features in the photomicrographs are presented here. Tensile testing of the flat specimens prepared by following ASTM standard (ASTM: Vol.03.01: E8M-00) was conducted in a computer controlled Instron universal (Model: 4204) testing machine using a cross head velocity of 0.5 mm/min. Values of yield strength (YS), ultimate tensile strength (UTS), uniform elongation (UEL), total elongation (TEL) and yield ratio (YR) were obtained from the numerical values of the machine output. The numerical output of the Instron universal testing machine has been tabulated in Table 2.

The hot rolled samples with 750°C FRT followed by air cooling and water quenching were subjected to isochronal (1 h) ageing treatment at different temperatures and the ageing behavior was studied by measurement of Vickers hardness values using a Brinell cum Vickers Hardness tester (Model: BV-250 (SPL)). The average hardness values with a maximum scatter of 3 – 5% for different samples are presented in Table 3. Subsequently, both the Cu - added and Cu - less samples, subjected to air cooling and water quenching after hot rolling with a FRT value of 750°C and provided with a 15% cold working, were subjected to ageing treatment. The ageing behavior of the samples was studied by following the similar procedure as was followed in

<table>
<thead>
<tr>
<th>Table 1</th>
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<tbody>
<tr>
<td>CHEMICAL COMPOSITION OF THE INVESTIGATED STEELS (Wt. %)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Steel Code</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>S</th>
<th>P</th>
<th>Ti</th>
<th>B</th>
<th>Cu</th>
<th>Fe</th>
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</thead>
<tbody>
<tr>
<td>S₀.₀</td>
<td>0.0845</td>
<td>1.303</td>
<td>0.035</td>
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<td>S₁.₅</td>
<td>0.0547</td>
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<td>0.529</td>
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<td>0.0436</td>
<td>0.0045</td>
<td>1.5230</td>
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Table 2
PROCESSING SCHEDULES AND MEASURED VALUES OF MECHANICAL PROPERTIES OF SAMPLES

<table>
<thead>
<tr>
<th>Steel Code</th>
<th>Mode of cooling after hot rolling</th>
<th>Mechanical properties</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td>YS (MPa)</td>
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<tr>
<td>S0.0</td>
<td>AC</td>
<td>298.3</td>
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<tr>
<td>S1.5</td>
<td>AC</td>
<td>350.5</td>
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<tr>
<td>S0.0</td>
<td>WQ</td>
<td>373.3</td>
</tr>
<tr>
<td>S1.5</td>
<td>WQ</td>
<td>490.1</td>
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</table>

Table 3
HARDNESS VALUES OF THE Cu – ADDED SAMPLES AT DIFFERENT AGEING TEMPERATURES

<table>
<thead>
<tr>
<th>Steel Code</th>
<th>Average hardness (VHN) of the hot rolled samples</th>
<th>Temperature of ageing (°C)</th>
<th>Average hardness (VHN) after ageing</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>AC WQ</td>
<td>AC WQ</td>
<td></td>
</tr>
<tr>
<td>S1.5</td>
<td>185 298</td>
<td>400 212</td>
<td>286</td>
</tr>
<tr>
<td></td>
<td></td>
<td>450 204</td>
<td>272</td>
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<td></td>
<td></td>
<td>500 235</td>
<td>302</td>
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<td></td>
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<td>265</td>
</tr>
<tr>
<td></td>
<td></td>
<td>600 222</td>
<td>250</td>
</tr>
</tbody>
</table>

the case of hot rolled samples and the results obtained have been presented in Table 4 with a maximum scatter between 3–5%.

3. RESULTS AND DISCUSSION

3.1 Effect of Cu Addition on the Transformation of Austenite

Figure 1 reveals microstructure obtained after completion of hot rolling at 750°C followed by air cooling in the case of Cu – less sample. It is apparent from Fig. 1 that the major microstructural constituent is polygonal ferrite (α) grain of uniform size. Occasionally, some phase aggregates are revealed along the boundary of the ferrite regions, which are

![SEM micrograph of air cooled sample (S0.0) showing polygonal ferrite grains of uniform size as the major microstructural constituent.](image-url)
apparently different from the appearance of martensitic islands usually observed in the microstructure of DP steels\textsuperscript{19}. Figure 2 presents the SEM micrograph of \textit{Cu} – added samples cooled in air after completion of rolling at 750°C. The microstructure (Fig. 2) contains a significant density of martensite islands distributed uniformly all over the ferritic matrix. The appearance, morphology and distribution of the second phase revealed in Fig. 2 are undoubtedly comparable with the martensitic islands observed in the DP microstructures presented in the literatures\textsuperscript{19}. Although the majority of ferrite grains appear polygonal, some elongated ferrite grains are clearly observed. The same micrograph also reveals martensite islands formed along the grain boundary of ferrite as well as within the ferrite grains without any observable interconnectivity between the islands. Figure 3 demonstrates the microstructural evolution obtained by carrying out water quenching of the \textit{Cu} – less samples after hot rolling. The microstructure essentially comprises polygonal ferrite grains and regions of two phase aggregates resembling upper bainitic microstructure. It is apparent that the polygonal ferritic grains are formed in the intercritical regions, while the two phase aggregates are the transformation products of austenite (\(\gamma\)) subjected to deformation in the intercritical region and subsequently quenched at a rate high enough to suppress the pearlitic transformation. On the other hand, the microstructure

<table>
<thead>
<tr>
<th>Steel Code</th>
<th>Average hardness (VHN) after 15% cold working (CW)</th>
<th>Temperature of ageing ((^\circ)C)</th>
<th>Average hardness (VHN) after ageing AC + CW</th>
<th>WQ + CW</th>
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<tbody>
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<td>S(_{1.5})</td>
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<td>200</td>
<td>239 318</td>
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<tr>
<td></td>
<td></td>
<td>250</td>
<td>251 329</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>300</td>
<td>275 323</td>
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<td></td>
<td></td>
<td>500</td>
<td>248 319</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 2: SEM micrograph of the sample cooled in air (S\(_{1.5}\)) showing significant density of martensite islands distributed uniformly all over the ferritic matrix.

Fig. 3: SEM micrograph of water quenched sample (S\(_{0.0}\)) showing the polygonal ferrite grains and regions of two phase aggregate resembling upper bainitic microstructure.
as shown in Fig. 4 formed by water quenching of the hot rolled Cu – added steel reveals a predominantly bainitic microstructure. It is also evident that the upper bainitic sheaves have formed randomly without any noticeable impression of the prior austenitic grain boundaries prompting to postulate that the defect regions in the austenite have acted as the potential sites for the nucleation of bainitic sheaves. Analysis of the microstructures shown in Figs. 1 – 4 envisages the inability of the present Ti, B microalloyed steel to form a ferrite – martensite DP microstructure by either air cooling or water quenching. In contrast, addition of 1.5 wt % Cu in the same steel has improved the hardenability of the same steel to an extent such that the ferrite – martensite DP microstructure is achieved by carrying out air cooling of the hot rolled steel. Such a variation in the trend of microstructure formation may be explained on the basis of the hypothesis that Cu aids in retarding the \( \gamma \rightarrow \alpha \) transformation \(^{16,17}\) in the present steel, and hence allows the austenite to remain enriched in terms of the partitioning elements. This in turn, has effectively stabilised austenite down to the \( M_s \) temperature in the case of the present steel. However, in the case of the water quenched samples, in the absence of formation of any ferrite in the intercritical region, the deformed austenite is stabilised so much so that it can resist the nucleation of any allotriomorphic ferrite in the grain boundary or in the defect regions. However, the nucleation of the bainitic ferrite is triggered as soon as the necessary undercooling is attained well above the \( M_s \) temperature. It may also be recorded here that the microstructure shown in Fig. 4 is reasonably comparable to the same formed in the high strength bainitic HSLA steels say, HSLA–80\(^{10,20}\), or HSLA–100\(^{10,21}\).

### 3.2 Mechanical Properties of the Air Cooled and Water Quenched Steels

The mechanical properties of the samples obtained after air cooling and water quenching of the hot rolled samples are presented in Table 2 and Figs. 5(a) and 5(b). It is evident that the YS, UTS, UEL and TEL values of the Cu - less air cooled steels are well comparable to the lower strength categories of low alloyed steels e.g., highly deformable DP steels. It is interesting to note that addition of Cu has significantly improved the YS and UTS values.
(Fig. 5(a)) in comparison to the Cu-less steel with a concomitant reduction in the UEL, TEL values (Fig. 5(b)), which are in conformance to the microstructural evolution of the concerned steels. Not only the YS and UTS envisage the advantage of Cu addition in the present steel, but also the YR value of the Cu – added samples (Fig. 5(a)) appears to be most favourable in spite of the observed reduction in the ductility values. The observed differences of the mechanical properties between the Cu – less and Cu – added air cooled samples may be substantiated by the absence of martensitic phase in the former and reasonable density of the same in the latter samples which resembles the distribution and morphology of the same commonly observed in classical DP steels.

In the case of Cu-less water quenched steels the observed mechanical properties may reasonably be compared with the HSLA-80 type of steel. The observed UTS and YS values suggest that the two-phase regions shown in Fig. 3 are presumably bainitic in nature rather than the fine pearlitic colonies. Table 2 shows that addition of Cu in water quenched steels has improved the YS and UTS values by a magnitude comparable to that of air cooled steels but with a lesser degree of concomitant detriment in ductility. The level of mechanical properties achieved in the Cu-added water quenched samples is comparable to the HSLA-100 class of steels.

The observed difference in the microstructural evolution (Figs. 3 and 4) and the mechanical properties between the Cu–added and Cu–less steels (Table 2) have substantiated the role played by Cu and B individually and in combination in retarding the \( \gamma \rightarrow \alpha \) transformation in the intercritical regions as well as in increasing the bainitic hardenability of \( \gamma \) during its subsequent transformation.

### 3.3 Ageing Behavior of the Cu – Added Steels

#### 3.3.1 Ageing Behavior of Air Cooled and Water Quenched Steels

The hardness values obtained by carrying out isochronal ageing treatment of the hot rolled samples at different temperatures are plotted in Fig. 6 with appropriate error bars to represent the percent scatter of data (~5%) for about 10 number of measurements at each ageing temperature. It is apparent that, the results shown in Table 3 and Fig. 6 exhibit that ageing treatment has improved the hardness of the air cooled samples particularly at the peak aged condition in contrast to the nominal softening of the water quenched samples. The observed improvement in hardness after ageing in comparison to the unaged samples in the case of air cooling may presumably be due to the fact that reasonable amount of Cu along with nominal amount of microalloying elements remained dissolved in austenite even after air cooling of the hot rolled steels which have subsequently precipitated during ageing. On the other hand, absence of any notable ageing response in the water quenched samples may presumably be due to occurrence of heterogeneous precipitation of phases containing Cu and other microalloying elements prior to ageing facilitated by higher density of interfacial area and smaller diffusion distance in bainitic microstructure. The disappearance of the martensitic islands after peak ageing of the air cooled samples as revealed in Fig. 7 is indicative of reasonable tempering of the same at the peak ageing temperature recorded i.e., 500°C. However, in spite of the disappearance of the martensitic islands reasonable improvement in hardness has been achieved by carrying out the ageing treatment at 500°C. Such an improvement in hardness may be attributed to the formation of precipitated phase appeared as small
bright spots in Fig. 7. It may be mentioned here that in the absence of appropriate characterization exercise, the exact identification of the precipitate phases remains beyond the scope of the present study. On the other hand, microstructural changes occurred during ageing of the bainitic sample at 500ºC and the consequent softening may presumably be presumably reduced the hardening effect of as quenched bainitic microstructure (298 VHN) to the extent such that ultimate softening has occurred after ageing in spite of the nominal hardening effect due to the formation of the precipitate phases during ageing.

3.3.2 Ageing Behavior of the Air Cooled Samples Subjected to 15% Cold Rolling

The microstructures of the air cooled samples subjected to 15% cold rolling and subsequent ageing treatment at 300ºC are shown in Figs. 9 and 10 respectively. The elongated morphology of the ferrite grains shown in Fig. 9 bears the signature of prior deformation. It is also apparent that the ferrite grains contain the dispersion of some faintly revealed tiny white spots occurred due to the formation of precipitate phases. The same micrograph (Fig. 9) further indicates that the martensitic regions have undergone a concomitant change in morphology identified as white regions with flat and feathery morphology in contrast to the isolated islands of martensite obtained in hot rolled and air cooled sample (Fig. 2).

The hardness values obtained after carrying out ageing treatment of the samples subjected to 15% cold rolling after air cooling of the hot rolled steels are presented in Table 4 and appended in Fig. 11. Suitable error bars have been appended in Fig. 11 interpreted as the result of tempering of bainite\textsuperscript{24} without the occurrence of any reasonable precipitation during ageing. It has earlier been demonstrated that the recrystallization and/or coarsening of bainitic sub units during tempering may effectively reduce the hardness value\textsuperscript{24}. Appearance of the approximately equiaxed ferritic regions in Fig. 8 after ageing at 500ºC is indicative of tempering of bainite. The tempering effect of bainite has

![Fig. 7: SEM micrograph of peak aged air cooled sample (S1.5) showing the tempering of the martensitic islands. Uniform dispersion of tiny white spots correspond to the formation of precipitate phases.](image)

![Fig. 8: SEM micrograph of peak aged water quenched sample (S1.3) showing the appearance of the approximately equiaxed ferritic regions formed due to the tempering of bainite during ageing.](image)

![Fig. 9: SEM micrograph of air cooled sample (S1.4) subjected to 15% cold working showing directionally elongated ferrite grains with reasonable dispersion of tiny white spots corresponding to the formation of precipitate phases. Martensitic regions have been identified as flat and feathery white regions.](image)
along with the individual plots following the similar procedure as has already been described in the case of Fig. 6. It is evident that the air cooled samples exhibit a significant improvement in hardness after cold working as well as after the ageing of the cold worked samples (Table 3 vis-à-vis Table 4) in comparison to the hardness of the air cooled samples. The observed increase in hardness may be attributed to the enhanced precipitation due to the presence of the defect structures formed during cold rolling. Even in the absence of appropriate microstructural evidence in the present study, it will not be unreasonable to predict that the defect structure and/or the defect precipitate complex may provide reasonable amount of hardening against the deformation during the indentation which has been manifested in terms of the improvement in hardness (185 VHN to 231 VHN) after cold working of the samples. It is also encouraging to note that the peak ageing has occurred at 300°C indicating an accelerated ageing response provided by the prior cold deformation. It may be assumed that the observed lowering of the peak ageing temperature after cold working has allowed to avoid any significant tempering of martensite as is evident in Fig. 10 by the presence of adequate amount of martensitic islands in the micrograph. It is also interesting to note that the density of the tiny white spots has reasonable increased in Fig. 10 in comparison to those in Fig. 9, which substantiates the fact that the deformation induced microstructural heterogeneities has essentially favored the formation of the precipitate particles resulting into significant hardening of the DP microstructure at a reduced level of ageing temperature.

3.3.3 Ageing Behavior of the Water Quenched Samples Subjected to 15% Cold Rolling

Figure 12 reveals the microstructure evolutions in hot rolled water quenched sample after 15% cold rolling. It is clearly evident that the parallel along with the individual plots following the similar procedure as has already been described in the case of Fig. 6. It is evident that the air cooled samples exhibit a significant improvement in hardness after cold working as well as after the ageing of the cold worked samples (Table 3 vis-à-vis Table 4) in comparison to the hardness of the air cooled samples. The observed increase in hardness may be attributed to the enhanced precipitation due to the presence of the defect structures formed during cold rolling. Even in the absence of appropriate microstructural evidence in the present study, it will not be unreasonable to predict that the defect structure and/or the defect precipitate complex may provide reasonable amount of hardening against the deformation during the indentation which has been manifested in terms of the improvement in hardness (185 VHN to 231 VHN) after cold working of the samples. It is also encouraging to note that the peak ageing has occurred at 300°C indicating an accelerated ageing response provided by the prior cold deformation. It may be assumed that the observed lowering of the peak ageing temperature after cold working has allowed to avoid any significant tempering of martensite as is evident in Fig. 10 by the presence of adequate amount of martensitic islands in the micrograph. It is also interesting to note that the density of the tiny white spots has reasonable increased in Fig. 10 in comparison to those in Fig. 9, which substantiates the fact that the deformation induced microstructural heterogeneities has essentially favored the formation of the precipitate particles resulting into significant hardening of the DP microstructure at a reduced level of ageing temperature.

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Figure 12 reveals the microstructure evolutions in hot rolled water quenched sample after 15% cold rolling. It is clearly evident that the parallel...
arrangement of the sub units in the bainitic regions (Fig. 4) is apparently absent in the micrograph of cold worked sample (Fig. 12). The cold deformed microstructure (Fig. 12) also reveals the appearance of thick white layers in the interferritic regions which may be attributed to the concentration of deformation induced inhomogeneities at the boundaries of deformed ferrite grains. Moreover, the microstructure reveals the appearance of well dispersed tiny white spots indicative of reasonable amount of precipitation in those regions.

The cold worked samples were subsequently subjected to the ageing treatments at different temperatures. The results obtained after ageing of the cold worked samples are shown in Table 4 and are appended in Fig. 11. It may be noted here that the water quenched samples have yielded nominal improvement in hardness in comparison to that achieved in the case of the air cooled samples (Table 3 vis-à-vis Table 4) within the range of ageing temperature. It is interesting to note that the peak ageing has occurred at 300ºC for air cooled samples and at 250ºC for water quenched samples. In spite of a lower value of peak ageing temperature observed in the case of the water quenched samples, the microstructure of the same as shown in Fig. 13 reveals a prominent tendency for coarsening of ferrite plates tending towards an approximate equiaxed morphology comparable to that observed in Fig. 8. Further thickening of the white regions (Fig. 12 vis-à-vis Fig. 13) may be due to further concentration of the deformation induced structural inhomogeneities in the interferritic regions. The observed difference in ageing behavior of the cold deformed air cooled and water quenched samples may be envisaged in terms of the much higher volume fraction of the grain boundary/interfacial regions in the water quenched samples (bainitic microstructure) than the air cooled samples (DP microstructure). The observed hardening effect at selected range of tempering temperature in the case of cold worked samples is comparable to the hardening effect usually achieved during the baking treatment adopted for painting the automobile body popularly known as bake hardening treatment.

Therefore it is apparent that the reasonably guessed postulation in the present investigation has been substantiated by the observation that ageing, particularly after nominal cold working, has yielded a remarkable hardening effect of DP microstructure and a moderate hardening effect for the bainitic microstructure at reasonably reduced values of peak ageing temperature under the present isochronal condition. It may be mentioned here that the investigations concerning the identification of the precipitate phases and determination of the precipitation kinetics of the Ti, B and Cu rich phases are in progress.

4. CONCLUSIONS

The results obtained from the present investigation have allowed to conclude the following:

i) The Ti-B microalloyed steel does not yield ferrite – martensite dual phase microstructure after subjecting the hot rolled samples to the selected cooling rates.

ii) After alloying of the Ti-B microalloyed steel with 1.5 wt% Cu, a dual phase structure is evolved by air cooling of the hot rolled sample with superior strength properties than the conventional dual phase steel with a concomitant lowering of the elongation values, which however is adequate for the purpose of DP steels.

iii) The Cu - alloyed Ti-B microalloyed steel has formed predominantly bainitic structure after water quenching, with the strength and
elongation values comparable to the HSLA-100 steels.

iv) Ageing treatments of the hot rolled samples do not respond much in improving the mechanical properties, rather at lower range of ageing temperature a notable tendency of softening has been observed in the case of water quenched sample.

v) 15% cold working of the hot rolled sample has nominally improved the hardness value for both the samples.

vi) Ageing treatment of the 15% cold deformed sample has resulted into significant age hardening effect at a range of temperature comparable to the conventional bake hardening temperature range.

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REFERENCES