Microstructural and micro hardness variation during low temperature recovery annealing in C-Mn high strength Steels

S Janakiram¹, Jai Gautam¹, P Sudharshan Phani² and Leo A I Kestens³

¹ School of Engineering Sciences and Technology, University of Hyderabad, India
² International Advanced Research Centre for Powder Metallurgy and New Materials (ARCI) Hyderabad India
³ Department of Electromechanical Systems and Materials, Ghent University, Ghent, Belgium

Email: sabavath.janakiram@gmail.com

Abstract Recovery was studied in four different grades of steel, ULC, HSLA and DP600 and DP800 with varying C-Mn content. All the steels samples were 80% cold rolled and annealed at 300 and 500°C for 30, 60, 120, 300 minutes, mainly to study the hardness and microstructural changes. Specimens were further tested using micro Vickers hardness tester. Microstructural changes were observed using scanning electron microscope. The cold rolled microstructure consisted of ferrite in ULC and ferrite-pearlite in HSLA, DP600 and DP800 steels. All the grains were pancaked and aligned in the rolling direction. The Hardness values increases for annealed specimens at 300 and 500°C in HSLA, DP600 and DP800 compared to cold rolled condition, which can be attributed to hardening of the ferrite matrix due to pinning of dislocations by carbon atoms. The hardness values for annealed specimens in ULC and DP800 decreases at 300 and 500°C which is attributed to softening.

1. Introduction

High Strength steels like HSLA and DP containing carbon and manganese are commonly used in automotive application and may undergo different thermo-mechanical processing routes, including hot rolling and cold rolling followed by annealing (recovery, recrystallization and phase transformation). The microstructure of C-Mn steels consists of higher volume fraction of ferrite and pearlite colonies (second phase) as remaining constituent. Pearlite colonies are composed of lamellar layers of cementite and ferrite. Carbon is mainly added to increase the strength, which segregates in the form of cementite in the pearlite colonies. C-Mn steels are cold rolled and annealed in the intercritical temperature to transform the initial ferrite pearlite microstructure to a final ferrite-pearlite microstructure [1]. Competition between recovery and recrystallization was studied in terms of hardness. Hardness decreases due to recovery and recrystallization and increases upon phase transformation from the initial cold rolled condition [1]. E.g. increase in hardness was reported in severe plastic deformed low carbon steels, with 0.05% C, in the temperature range of 300-600°C for various soaking time [2]. Increase in yield strength was reported in DP and TRIP steels by pre straining from 2-10% and paint baking at 140-220°C for various soaking time [3-4] However, in the pearlitic steel consisting of 0.7% carbon it was reported that pearlite lamellae spheroidized, leading to softening mainly due to cementite dislocation.
interaction. The acceleration of spheroidization depends on the amount of cold working and annealing temperature [5].

In all the above-mentioned investigations special processing practices were adopted compared to the conventional ones with the purpose to enhance the strength of the material at different temperatures and soaking time. C-Mn steels with different C-Mn content were cold rolled and annealed in the intercritical temperature range were observed to increase in hardness compared to the cold rolled condition [6].

The present study was designed to investigate the effect of lower annealing temperature and soaking time to understand the recovery behaviour with variation in carbon and manganese in particular grades of high strength steels like HSLA, DP600 and DP800.

2. Experimental procedure:
The chemical composition of the steels used in this study is reported in table 1. Hot rolling of all the steels was carried out above the austenite recrystallization temperature and coiled at pearlite start temperature, to obtain ferrite-pearlite hot bands, which are further cold rolled to 80% reduction. From the above steels specimen were cut to 10 mm width and 12 mm length in the rolling direction. All the samples were heat-treated in muffle furnace at 300 and 500°C for various soaking time 30, 60, 120, 300 minutes soaking time followed by furnace cooling. These specimens were mechanically polished and etched with nital to study the microstructural changes using FESEM. Hardness values were recorded on flat well-polished samples without etching, at least 15 indents were recorded for each specimen with 500gm load and 20 s dwell time. Nanoindentation tests were performed using a commercially available nanoindenter iMicro® with an InForce50 actuator from Nanomechanics Inc., Oak Ridge, USA. Multiple large indentation maps with 2500 indents per map on a 50 x 50 mm² area was collected using the NanoBliz 3D high speed mapping technique. An indentation spacing 1 µm was maintained and the load was changed such that the indentation depth was approximately 100 nm, which results in a depth to spacing ratio of 10 based on the recommendation of [7].

| GRADE | C     | Mn     | Si    | Pearlite Area fraction | Cold Rolled Hardness |
|-------|-------|--------|-------|------------------------|----------------------|
| ULC   | 0.002 | 1.28   | 0.22  |                        | 255±5.2              |
| HSLA  | 0.06  | 1.32   | <0.008| 7.6± 0.5               | 268 ±5.8             |
| DP600 | 0.09  | 1.65   | 0.28  | 29.6± 6.2              | 277± 4.5             |
| DP800 | 0.13  | 2.00   | 0.25  | 33.5± 4.6              | 282± 5.9             |

3. Results

3.1. Cold rolled microstructure, morphology and hardness
Cold rolled microstructures consist of two phases, ferrite and pearlite, in all steel grades, except ULC steels which has only ferrite. With increasing amount of carbon, the amount of pearlite increases. Pearlite colonies consist of alternate lamellar layers of cementite and ferrite. The area fraction of pearlite in hot rolled condition is reported in table 1. The pearlite morphology in HSLA consists of lamellar, fragmented coarse and fine cementite particles at the interface after 80% cold rolling. With increasing amount of C-Mn in DP600 and DP800, pearlite appears in the form of bands and explained in [8]. The cementite lamellar morphology is retained in DP600 grade after 80% cold rolling, whereas the DP800 grade exhibits both lamellar and fragmented cementite morphology after cold rolling. As amount of carbon content increases the density of cementite increases, which leads to increases in the thickness of pearlite colony cf. figure 1, 30 min in HSLA, DP600 and DP800. With increase in carbon content the hardness values in the cold rolled condition increases from ULC to DP800 as shown in table 1.

3.2. Microstructure variation after low temperature recovery
3.2.1. Variation in microstructure at low temperature recovery 300°C

Microstructural variation after annealing at 300°C for different soaking time in four different grades of cold rolled steels are shown in figure 1. In three grades of steel the microstructure consists of ferrite (in grey) and a pearlite colony appears in white. In ULC, the cold rolled microstructure is retained and consisted of the ferrite phase only. In HSLA, for the first 30 min annealing the cold rolled microstructure is retained, but with increasing soaking time, at 120 min the dissolution of cementite takes place and continues with increasing soaking time up to 300 min. The dissolution of cementite in the ferrite matrix can be confirmed by the thinning of the cementite lamellae as indicated in the rectangular box as in figure 1, (300 min). In DP600, the cold rolled microstructure is retained till 120 min of soaking time, at 300 min fragmentation of cementite slowly appears as indicated in the rectangular box in figure 1. In DP800, no microstructural changes in ferrite nor pearlite are observed till 120 min of soaking. However, with increasing soaking time to 300 min, partial spherodization of pearlite lamellae takes place in the pearlite colonies figure 1, DP800, 300 min.

![Figure 1. Shows variation of microstructure in all the four alloys with temperature at (a) 300° for 30, 60, 120 and 300 min of soaking. F-ferrite, P-pearlite, Box indicating spherodization at ferrite-pearlite interphase](image)

3.2.2. Variation in microstructure at low temperature recovery 500°C

The microstructural variation with annealing at 500°C for various soaking time in four different grades of cold rolled steels are shown in figure 2. In ULC, recrystallization of ferrite takes after 30 min of soaking time, as indicated by the circle in figure 2 and continues to increase with increase in soaking time. In the HSLA steel, the pearlite colony remains the same for the first 30 min of soaking time, but with increase in soaking time spherodization of pearlite lamellae takes place. In DP600, the ferrite pearlite microstructure is retained till 30 min of soaking time, but with the increase in soaking time, the
complete lamellar structure is replaced by fine cementite particles (spheroidized) in the pearlite colony. In the DP800 grade, the lamellar cementite structure spheroidizes inside the pearlite colonies from first 30 min soaking time and continues with increase in time.

![Diagram](Image)

**Figure 2.** Variation in microstructure in all the four alloys at 500°C for 30, 60, 120 and 300 min of soaking time

### 3.3. Variation of hardness after low temperature recovery

#### 3.3.1. Variation of hardness after low temperature recovery 300 °C

Cold rolled sheets of different C-Mn content are annealed at low temperature 300 °C for different soaking time. The variation of hardness as a function of C-Mn content for different soaking time is shown in figure 3(a) and 3(c) corresponding increase and decrease in hardness. At low temperature 300°C, cf. figure 3(a), ULC steel hardness increases after first 30 min of soaking time and then decreases with increase in time. This decrease in hardness can be attributed to recovery in ULC. In HSLA steel, the hardness first increases with 30 min of soaking time, then slightly decreases at 60 min and saturated thereafter with increase in time. This increase in hardness is mainly due to increase in carbon content which leads to formation of pearlite phase, which was absent in the ULC. In DP600, hardness increases from cold rolled condition and is higher than HSLA steel as the area fraction of pearlite increases, cf. in table 1. The hardness increases with continuous increase in soaking time. In DP800, the hardness significantly increases till 120 min of soaking time in comparison with HSLA and DP600. With further increase in soaking time, the hardness decreases below the value of DP600 at 300 min, even though the area fraction of pearlite is higher than DP600. This decrease in hardness can be linked to morphological changes in DP800 as in figure 3(a), 300 min. It was also observed that the hardness is still higher as compared to the cold rolled condition. In summary, the hardness decreases in ULC steel from cold rolled
condition, whereas the hardness increases in HSLA, DP600 and DP800 with presence of ferrite and pearlite as second phase from initial cold rolled condition by recovery annealing at 300°C.

![Graph showing hardness variation](image)

**Figure 3.** Shows variation of hardness as a function of C-Mn content with temperature at (a) 300°C and (b) 500°C, percentage increase and decrease of correspond Hardness variation (c-d)

3.3.2. **Variation of hardness after high temperature recovery 500°C**

At high temperature recovery 500°C, cf. figure 3(b) and (d), both increase and decrease in hardness are observed. In ULC steels, the hardness drop significantly and continues to decrease with increasing soaking time, which is attributed to recrystallization and grain growth. In HSLA steel, the hardness increases first for 30 min and then starts decreasing below the value corresponding to the cold rolled condition with increase in soaking time. The DP600 grade shows the similar kind of behaviour as HSLA for first 30 min. It drops back to the cold rolled hardness condition at 60 min and decreases further with increasing soaking time. In the DP800 grade, the hardness starts decreasing below the cold rolled condition after annealing for 30 min and continues decreasing thereafter with increasing soaking time. In summary, the hardness starts to decrease below the initial cold rolled condition in ULC and DP800 steel, right away after 30 min of soaking time. Alternatively, for grades HSLA and DP600 first the hardness increases for 30 min and the starts decreasing below the cold rolled condition.

3.4. **Nano indentation mapping**
As shown in figure 4, further investigation was carried out by nano indentation mapping showing variation in hardness from cold rolled condition to annealing at 300 and 500°C for 30 min in DP800. This results further confirms microstructure consisting of ferrite and pearlite in DP800, hardness increase from cold rolled condition to low temperature annealing at 300°C, the peak shifts to right side showing increase hardness as in figure 4(d) and reaches to its initial cold rolled condition after annealing at 500°C as in figure 4(d). It can be clearly seen that hardness increases in the pearlite colony and ferrite-pearlite interface.

![Figure 4](image)

**Figure 4.** Variation in hardness form Nano indentation mapping in DP8 (a) cold rolled (b) 300°C (c) 500°C for 30min soaking time (d) pixel counts vs hardness map

4. Discussions

Recovery at low temperature by varying C-Mn content, resulted in microstructural and hardness variation occurring in four different grades of steels. Increasing the amount of C-content, the microstructure changes from ferrite to ferrite-pearlite. The changing pearlite area fraction resulted in different cementite morphology after 80% cold rolling, which resulted in microstructural and hardness variation after low temperatures recovery treatment. In ULC steels only the ferrite phase is present due to the minute amount of carbon and manganese. The hardness slightly decreases with increasing soaking time due to recovery. Additionally, the hardness significantly drops below the cold rolled hardness due to recrystallization at 500°C. From the microstructural analysis it was observed that the cold rolled microstructure is retained but hardness increases compared to the cold rolled condition on annealing at 300°C in HSLA, DP600 and DP800. This increase in hardness of C-Mn steels can be related to the strengthening mechanisms applicable to the bake hardening typical in low carbon steels after small cold rolling, which is essentially a strain aging phenomenon, whereby interstitial carbon atoms pin the mobile dislocations and form the Cottrell atmosphere associated with first stage of hardening in addition the precipitation of carbide would also lead to increase in yield strength as a second stage of bake hardening reported by [9]. As the carbon content in ULC steels is very low therefore least amount of carbon is available to pin the dislocation and henceforth only softening, recovery and recrystallization occur with increase in temperature from 300 to 500°C. Increase in amount of C-content where microstructure change from ferrite to ferrite-pearlite. Cold rolling to 80% leads to excess of dislocations mainly in pearlite colonies, at ferrite-pearlite interfaces and inside ferrite grains like happening during severe plastic deformation reported in [10]. The amount of dislocations is relatively higher inside the pearlite colony compared to inside the ferrite grain [10]. This increase in dislocation density by cold rolling and availability of more carbon atoms by increase C-content resulted in an increase of hardness by pinning of mobile dislocation. This was evident from the nano indentation mapping; in the cold rolled condition the hardness was higher in the pearlite colony than in the ferrite as in figure 4(a). With low temperature annealing, cf. figure 4(b) the hardness increases in the pearlite colony at the interface and inside the ferrite grains, which can be attributed to pinning of the dislocation by precipitation of carbon atoms. Increasing the amount of carbon in HSLA, DP600 and DP800 causes the area fraction of pearlite to increase and hardness keeps increasing inside the ferrite grain, in the pearlite colonies and at the ferrite-pearlite interface as well. The increase in hardness can also be due to pinning of the dislocations by the
second phase particles like Fe₃C nitrides and carbonitrides [11]. This increase in hardness needs to be further investigated and confirmed by TEM studies. After recovery annealing at 500°C, it has been observed that retention of the cold rolled microstructure leads to an increase in hardness in HSLA and DP600 for the first 30 min of soaking time. With the increase in soaking time the lamellar structure is replaced by fine spherical particles (spherodization) and the hardness decreases below the cold rolled condition with increase in time. In DP800 steel, the hardness starts decreasing from the first 30 min of soaking time due to spherodization of cementite. It is well documented in the literature that spherodization of cementite leads to softening, which is in good agreement with the present result [5]. It was observed that hardness decreases in HSLA steel for 120 and 300 min of soaking time, in DP800 grade at 300 min of soaking time. This decrease in hardness can be attributed to softening taking place due to partial spheroidization. However, the hardness still remains higher than the cold rolled hardness, which can be linked to the microstructure in the pearlite colony, which consists of a combination of lamellar, fragmented and partial spheroidized cementite lamellae. The lamellar and fragmented cementite contributes to the increase in hardness, whereas the spheroidization leads to a decrease in hardness. However, the question arises why does pearlite lamellae spheroidize at 300°C. It is has been reported that acceleration of spheroidization is mainly due to a higher amount of plastic straining and annealing temperature [5]. In DP800, consisting of 34% of pearlite and due to 80% cold rolling, the interlamellar spacing of cementite decreases and becomes thinner and accelerates the spheroidization by cementite dislocation interaction phenomena [12]. Due to finer and fragmented cementite morphology rapid spheroidization takes place at 300°C after 30 min of soaking time. In a recent in-situ study under synchrotron in dual phase steels reported that dislocation of annihilation rate is almost negligible at 300C and softening starts at 500C [13].

5. Conclusions
The present results show the significant effect of carbon on variation in microstructure and hardness in C-Mn high strength steels containing pearlite and the following conclusions can be drawn

- In ULC steel, where the less amount of C-Mn is present, and hence the microstructures consists of a single ferrite phase, softening takes place and leads to normal recovery at 300°C and recrystallization at 500°C.
- In HSLA, DP600 and DP800 grades, the increasing pearlite content resulted in an increase in hardness after recovery annealing at 300°C. The cold rolled microstructure is retained and hardening takes place in the microstructure.
- Softening takes place with increase in annealing temperature to 500°C and increase in soaking time due to spheroidization of cementite.
- Nano indentation mapping confirms the hardness increase inside ferrite grain, pearlite colonies and at the ferrite-pearlite interface due to precipitation of carbon atoms and pinning of dislocation.

6. References
[1] Peranio N, Li Y J, Roters F and Raabe D 2010 Materials Science and Engineering A 527 4161–4168
[2] Ghiabakloo H and Kazeminezhad M 2017 Met. Mater. Int. 23 5984-993
[3] Ramazani A, Bruehl S, Gerber T, Bleck W and Prahl U 2014 Materials and Design 57 479–486
[4] Timokhina I B, Hodgson P D, Ringer S P, Zheng R K and Pereloma E V 2009 steel research Int 80 7
[5] Chojnowski E A and MeG. Tegart W J 1968 Metal Science Journal Vol 2
[6] Janakiram S, Gautam J and Leo kestens A I 2019 manuscript submitted at ReX GG IOP conference series (under review)
[7] Oliver WC and Pharr GM. 1992 J Mat Res. 7 1564-83.
[8] Janakiram S, Gautam J, Miroux A Moerman J and Leo Kestens A I 2019 Diffusion Foundations 22 84-93
[9] De A K, Vandeputte S, Soenen B and De Cooman B C 2001 Scripta Materialia 44 695-697
[10] Shin D H, Kim Y S and Lavernia E J 2001 Acta mater 49 2387–2393
[11] Ahmet Bulbula and Ramazan Kaçara 2017 Materials Research 20 210-217
[12] Languillaume J, Kapelski G and Baudelet B 1997 Acta mater 45 3 1201-1212
[13] Marc Moreno, Julien Teixeira, Guillaume Geandier, Jean Christophe Hell, Frederic Bonnet, Mathieu and Sebastien Allain Y P 2019 Metals 9 1-13