

## Heat treatment and effects of Cr and Ni in low alloy steel

MOHAMMAD ABDUR RAZZAK

Materials and Metallurgical Engineering Department, Bangladesh University of Engineering and Technology (BUET), Bangladesh

MS received 1 March 2010; revised 14 February 2011

**Abstract.** The effects of Cr and Ni on low carbon steel was observed. Undissolved carbide particles refine the austenite grain size. In the presence of nickel, chromium carbide is less effective in austenite grain refinement than chromium carbide in absence of nickel at temperature below 975°C. Nickel does not produce any austenite grain refinement but presence of nickel promotes the formation of acicular ferrites. It was also found that Ni and Cr as chromium carbide also refines the ferrite grain size and morphology. Cr as chromium carbide is more effective in refining ferrite grain size than nickel.

**Keywords.** Low alloy steel; Cr; Ni.

### 1. Introduction

During the last fifty years, there has been a great demand for steels with higher tensile strength, adequate ductility and toughness. This has been particularly so where lightness is desirable, as in the automobile and aircraft industries. An increase in carbon content met this demand in a limited way, but even in the heat-treated condition the maximum strength is about 700 MPa above which value, a rapid fall in ductility and impact strength occurs and mass effects limit the permissible section (Aver 1974). Heat treated alloy steels provide high strength, high yield point, combined with appreciable ductility even in large sections. The use of plain carbon steels frequently necessitates water quenching accompanied by the danger of distortion and cracking, and only thin section can be hardened throughout. For resisting corrosion and oxidation at elevated temperatures, alloy steels are essential. The Alloy Steels Research Committee adopted the following definition: 'Carbon steels are regarded as steels containing not more than 0.5% manganese and 0.5% silicon, all other steels being regarded as alloy steels' (American Society for Metals 1964). The principal alloying elements added to steel in widely varying amounts either singly or in complex mixtures are nickel, chromium, manganese, molybdenum, vanadium, niobium, silicon and cobalt. The observation in this paper is directed towards experiment on low alloy steels (0.11 wt% C) which has a high weldability and toughness. These properties are in high priority for structural steel if these can be blended in with a high tensile strength. The sole purpose of this study is to find out proper alloying elements to increase the tensile properties with appropriate heat treatment.

In the current study four different steels with varying weight percentages of Ni and Cr were used to observe the effects of alloying elements. The effects were characterized according to the presence of alloying elements either alone or in conjunction with each other in the low carbon steel samples. The steel samples were characterized using mechanical testing method. Tensile strength and elongation data were collected to compare the effects of heat treatment and alloying elements. Metallographic analysis was also done to draw a correlation between alloying elements and microstructural feature like grain size and morphology.

### 2. Experimental

#### 2.1 Specimen preparation

Four different steels containing about 0.11% carbon were used in this study. The composition of the steels is presented in table 1. Steel 1 is the base steel with which the structure and properties of other steels 2–4 are compared. The steels were made previously in an air induction furnace in our department. About 14 mm diameter specimens were rolled down from 16 mm diameter bars of each of the steels in order to study the austenite grain coarsening behaviour.

#### 2.2 Carburization and measurement of austenite grain size

Since size of the austenite grains directly affect the subsequent structure and hence the properties of steels, a study was made to determine prior austenite grain size at temperatures higher than upper critical temperature.

(mohammad.razzak@insa-lyon.fr)

**Table 1.** Composition of the samples.

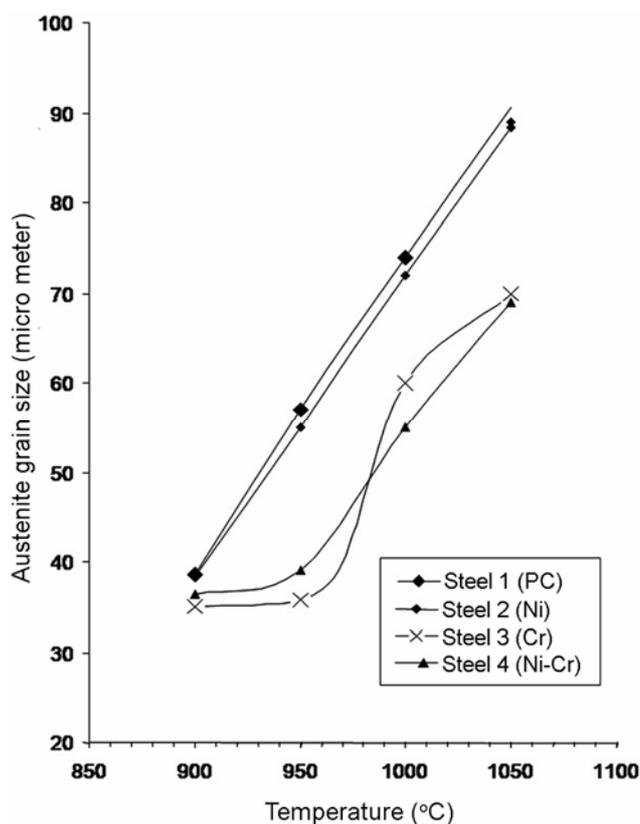
Steel no.	Composition (wt.%)						
	C	Si	Mn	S	P	Cr	Ni
1	0.10	0.28	1.18	0.021	0.028	–	–
2	0.13	0.16	1.31	0.027	0.038	–	1.26
3	0.11	0.20	1.28	0.022	0.022	0.5	–
4	0.11	0.20	1.28	0.022	0.022	0.69	0.97

Carburization technique was used to reveal prior austenite grain size. There are also other methods in determining prior austenite grain size like isothermal transformation technique, oxidation technique, etc. But previous work showed that the isothermal technique did not work well in revealing prior austenite grain boundary of low alloy steels (Haque 1989). So, carburization technique was adopted to reveal prior austenite grain boundaries of steels in this work.

The technique is based on the formation of a continuous cementite network at the austenite grain boundaries. Carbon will diffuse in steel from the carburizing atmosphere forming hypereutectoid steel at the surface of the specimen and during slow cooling in the furnace continuous cementite network is formed at the austenite grain boundaries at the selected austenitizing temperatures. Subsequent etching of the furnace cooled samples revealed the cementite network formed which marked the prior austenite grain size at the selected carburizing temperatures (Clark and Varney 1962).

Solid carburizing or pack carburizing technique was applied for this experiment. The steel specimens were heated to different austenitizing temperatures, i.e. 900–1050°C with an interval of 50°C. Before heating these specimens, they were packed in a pot with carburizing mixture. Then they were placed in Blue-M furnace. After reaching the desired temperature, they were held at that temperature for 2 h to reach near the equilibrium condition and then cooled in this furnace to room temperature. Slow cooling ensured a continuous cementite network through the austenite grain boundaries.

The assessment of prior austenite grain size was made from direct measurement of the austenite grains in the specimens under optical microscope. The grain size was measured using the mean linear intercept method, counting grain boundary intersections with the circumference of the circle in the eyepiece of a microscope. The effective circumference of the circle was determined precisely by measuring its diameters with reference to a stage micrometer at the magnification used. A total of at least 300–600 intersections were counted for each specimen. Then the size of austenite grain was measured using the mean linear method.

**Figure 1.** Variation of prior austenite grain size with temperatures for steels 1–4.

The heat-treatment temperatures of steels were determined by a careful examination of the austenite grain size. The criteria for the determination of the heat-treatment temperature of steels were that the steel had the same austenite grain size and that the temperatures were such that an appreciable proportion of the solute elements had entered into solution for subsequent precipitation. This is indicated in figure 1 by a steep rise of the austenite grain size. An austenite grain size of 40 µm was found to be suitable and the corresponding heat-treatment temperatures for the steels were 910, 910, 970 and 950°C for the steels 1–4, respectively.

Thermodynamic calculation was done using Matcalc using the database 'mc\_sample'. Calculation of results

presented in figure 2 show that chromium in the steels precipitated as  $M_3C_2$ . The dissolution temperatures obtained by the equilibrium thermodynamic calculation is below the one expected from the experimental one. As heat treatments were done for 2 h at higher temperatures, it is logical to expect that the precipitating elements will be completely in solution at the chosen heat treatment temperature.

### 2.3 Mechanical testing

The heat treated 13 mm diameter bar was then machined into standard tensile specimens with a nominal diameter and gauge length of 3.99 and 25 mm respectively. The grip size was chosen to be 13 mm in width and in length as shown in figure 3. The tensile specimens were then tested with a Universal Tensile Testing Machine (INSTRON) to obtain data on yield strength (YS), ultimate

tensile strength (UTS), percentage of elongation (% EL), and percentage of reduction in area (% RA).

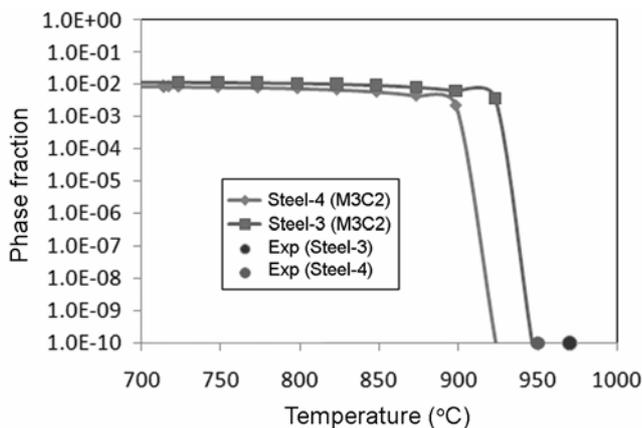
### 2.4 Optical microscopy of fracture samples

Samples from fractured tensile specimens were taken for microscopic examination. To avoid heavily deformed zone for microstructure observation, samples were chosen from the grip of the tensile test specimen as shown in figure 3. The samples were then ground, polished up to  $\gamma$ -aluminum powder and then etched in 5% Nital solution. The microstructure of these specimens was then studied. Optical microscope photograph (figure 4) of the microstructure of each specimen was taken to compare the microstructural features in conjunction with the mechanical properties.

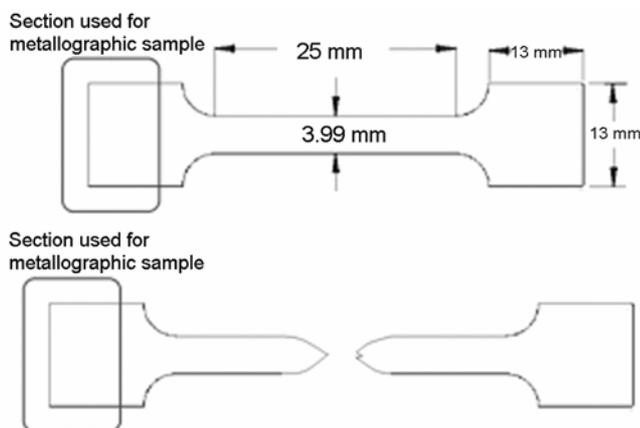
## 3. Results and discussion

The prior austenite grain size vs temperature graph is plotted and shown in figure 1. It can be observed from figure 1 that the austenite grain size increases with increasing austenitizing temperature. For steels 1 and 2 this relation is almost linear. Steel 1 is a plain carbon steel and it does not contain any alloying element. So there is no obstruction for grain growth and the austenite grain size increases rapidly and linearly with temperature. Steel 2 is basically steel 1 with nickel added to it. This steel also showed similar austenite grain coarsening behaviour as that of plain carbon steel 1. Nickel remained in solid solution and not combined with carbon to produce any second phase particles. In absence of second phase particles in steel 2, grain growth is not hindered.

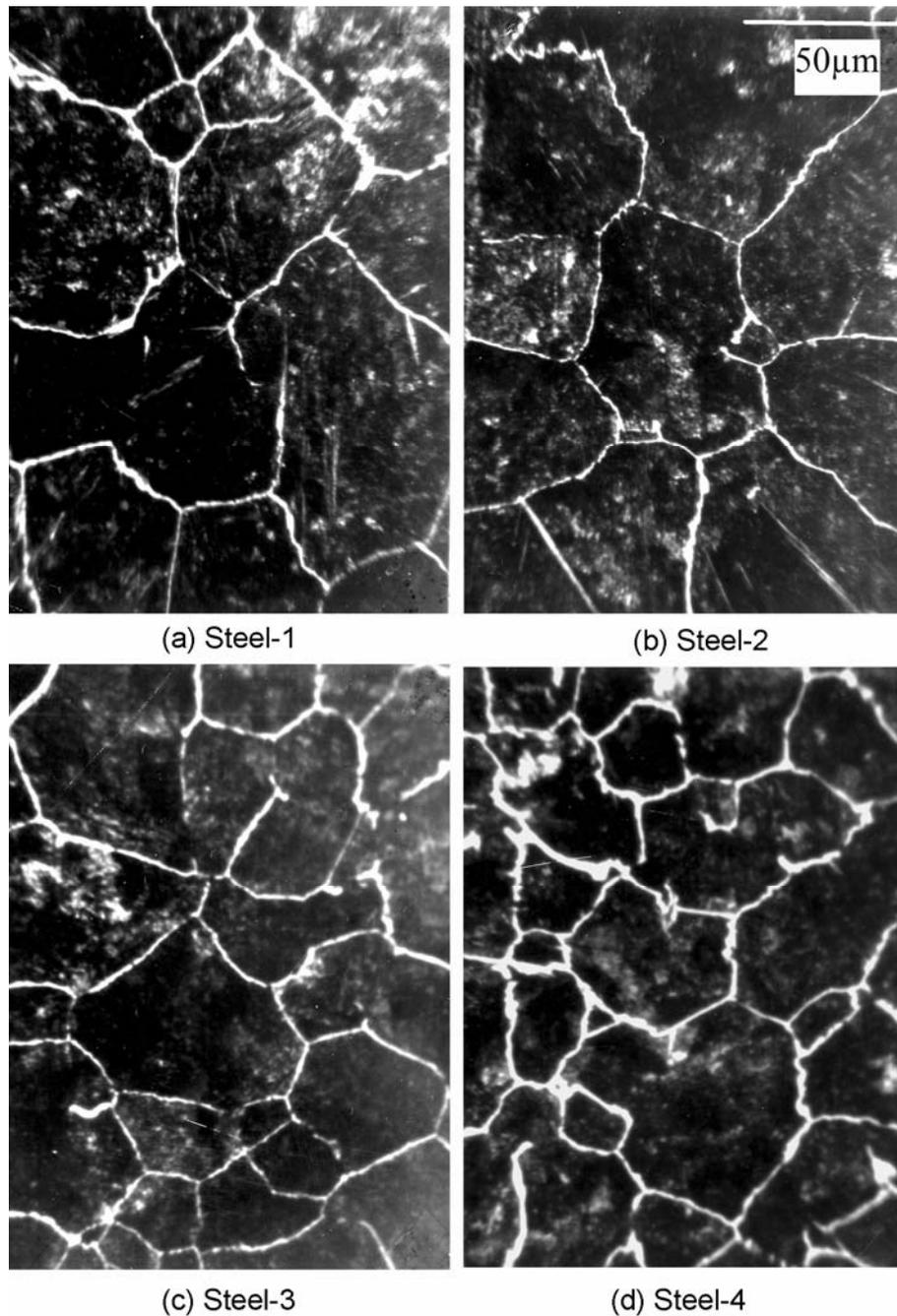
Steel 3 produced the finer austenite grain size than steel 1. Cr combines with carbon and forms chromium carbide precipitates (Aver 1974). These precipitates pin the austenite grain boundaries and inhibit grain growth resulting in finer austenite grain size than steel 1 (plain carbon). Steel 4 also produced grain size finer than steel 1 and coarser than steel 3 (Rollason 1961; Clark and Varney 1962; Aver 1974). Steel 4 contains both Cr and Ni. As can be seen from steel 1 nickel does not have any effect on the austenite grain refinement. So the finer austenite grain size in steel 4 compared to steel 1 is clearly due to the effect of chromium carbide precipitates. The austenite grain size of steel 3 remains finer up to 950°C beyond this temperature; there is a steep rise in austenite grain growth. Steel 3 produced finer grain size than steel 4 with (Ni + Cr) up to a temperature of 975°C and above this temperature steel 4 produced finer austenite grain size than steel 3. Composition of steel suggested that steel 3 (Cr = 0.69 wt.%) should contain slightly higher volume fraction of precipitates than steel 4 (Cr = 0.5 wt.%). Presence of higher volume fraction of precipitates increased the grain boundary pinning (Zener



**Figure 2.** Matcalc thermodynamic calculation results using mc\_sample database for steels 4 and 3. Evolution of phase fraction is plotted with temperature.



**Figure 3.** Dimensions of tensile specimen used for tensile test and location on sample for subsequent microstructure study is shown.

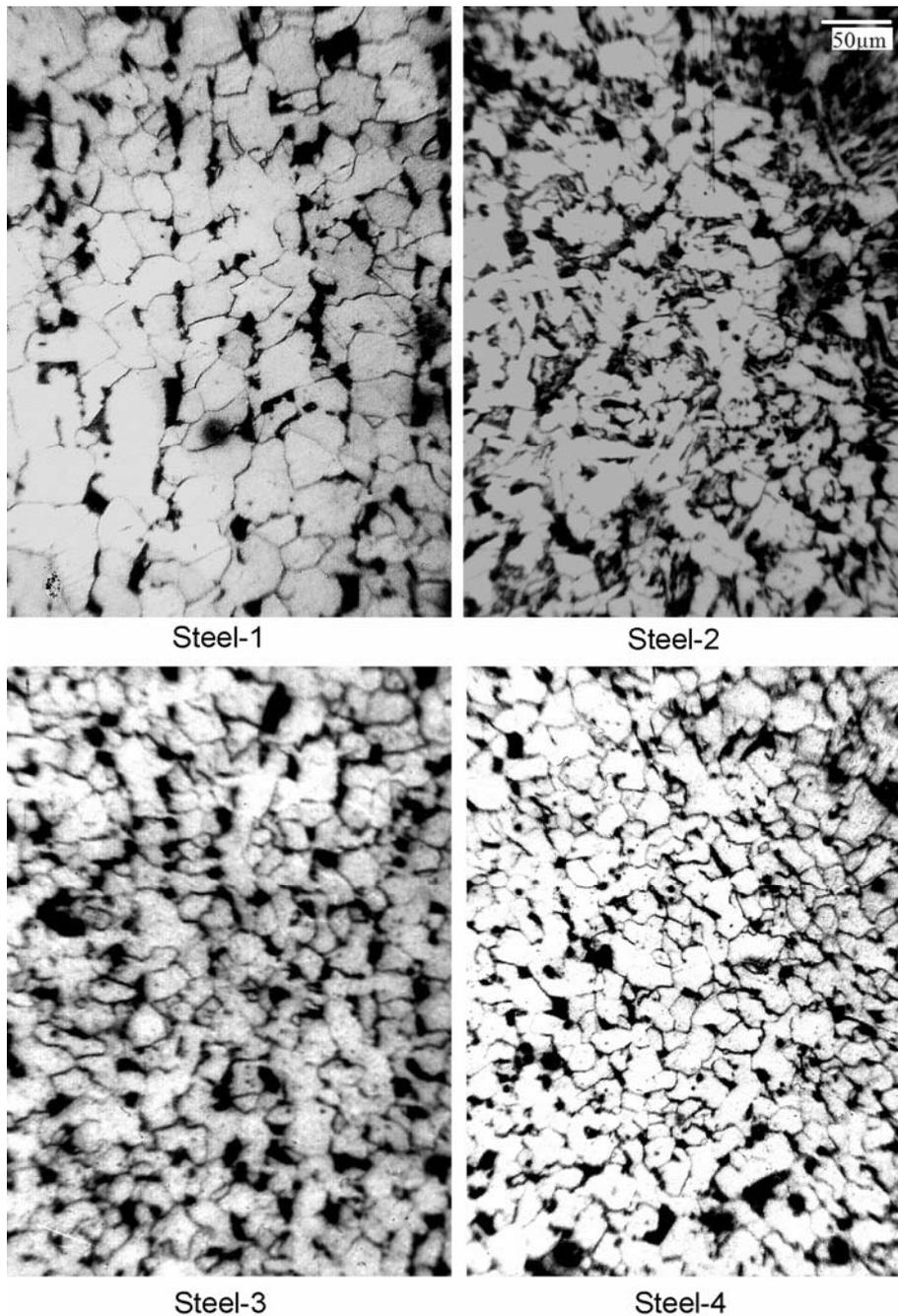


**Figure 4.** Optical micrograph showing prior austenite grain boundaries of steels 1–4 revealed by carburization technique at 1000°C ( $\times 200$ ).

pinning) efficiency and thus results in smaller grain size. The results also indicate that in presence of nickel, chromium carbide is less effective in austenite grain refinement than chromium carbide in absence of nickel at temperature up to 975°C. So, presence of nickel lowers the chromium carbide dissolution temperature in the low alloy steels. Among the three alloys, steel 2 showed the same grain coarsening behaviour as plain carbon steel. This confirms the previous understanding that Ni has no effect

on the austenite grain size refinement. Finer austenite grain size of steels 3 and 4 indicated that Cr as chromium carbide is an effective grain refining element for HSLA.

The microstructures of steels 1–4 cooled at 120°C/min are shown in figure 5. It was observed that steels 1–4 showed regular ferrite–pearlite structure with some ferrite morphology change in steel 2. The microstructure observed comprised of fine ferrite–pearlite structure where the pearlite is isolated in the ferritic matrix. Steel 2



**Figure 5.** Optical micrograph of steels 1–4 cooled at 120°C/min ( $\times 200$ ).

at the fast cooling rate of 120°C/min showed some widmanstatten ferrite along with regular ferrite pearlite. Steels 2–4 produced finer ferrite pearlite than plain carbon steel 1. Among the three alloys, steel 3 produced finer grain size than steel 2 and steel 4 produced the finest grain structure of all steels. Steel 2 has Ni as alloying elements; hence it showed smaller grains in comparison with the plain carbon steel 1. It is well known that Ni lowers the austenite to ferrite transformation temperature and thus produced condition for smaller ferritic grains to

nucleate at relatively low grain mobility condition. Besides that from the microstructural observation it was also clear that Ni modified the ferrite morphology to more acicular shape.

Steel 3 contains Cr. Cr combines with C and formed chromium carbide precipitates during cooling from the austenite zone. These chromium carbide precipitates pin the newly nucleated ferrite grain boundaries and thus fine ferrite grain is obtained. The finer grain size of steel 3 than steel 2 clearly indicate that Cr as chromium carbide

is more effective in ferrite grain size refinement than nickel. Steel 4 contains both Ni and Cr. The finest ferrite grain size of this steel is due to the combined effects of nickel and chromium. So it can be said that Cr in presence of Ni is more effective in producing finer microstructure.

In figure 6, average grain diameter of steels 1–4 is presented. Grain size measurement also suggested the same phenomenon predicted by microstructural observations. Considering the experimental error it can be said that steel 4 (Ni + Cr) has the smallest mean grain diameter of  $\sim 15 \mu\text{m}$ . Steel 2 showed smaller grain diameter than steel 1 but it should be considered that acicular morphology of ferrite in the microstructure of steel 2 produced greater extent of error in the measurement by mean linear intercept method. So ferrite grain size measurement for steel 2 is not fully reliable.

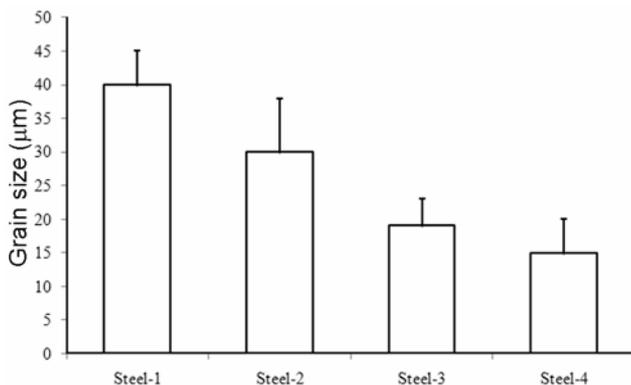
Tensile test results from the specimens of steels 1–4 cooled at  $120^\circ\text{C}/\text{min}$  are shown in figure 7. It is evident from the figure that yield strength of steels 2–4 is higher than the base steel 1 (PC). Among the three alloy steels, steel 4 with Ni and Cr produced the highest yield strength and steel 2 with Ni produced the lowest yield strength. Steel 3 with chromium produced yield strength in between steels 2 and 4. A similar trend was found with the ultimate tensile strength of these steels. The higher yield strength of steel 3 with Cr than steel 2 with Ni indicated that Cr is more effective than nickel in increasing yield strength. The highest yield strength of steel 4 with Ni and Cr is clearly due to the combined effects of Ni and Cr.

Nickel does not produce any second phase particle. Ni is found mostly in the form of solid solution in the ferrite (Rollason 1961; Clark and Varney 1962; Aver 1974). So Ni increased the strength of the steel by solid solution strengthening. Besides that Ni also lowers the transformation temperature (Rollason 1961; Clark and Varney 1962; Aver 1974), so the lower transformation temperature produces smaller ferrite grains. Besides that from the

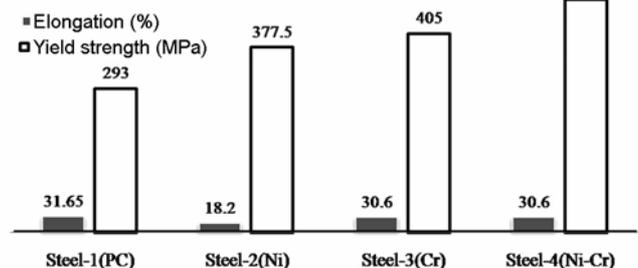
microstructural observation it is seen that presence of Ni promotes acicular ferrite formation. Change in morphology of the ferrite to acicular ones also produces obstacle in dislocation glide. Thus nickel increases the strength by refining the grains by lowering the transformation temperature and also changing the morphology of the ferrite grains.

Chromium in the form of chromium carbide precipitates increased the strength by means of precipitation strengthening. Secondary chromium carbides pin the grain boundaries and inhibit the grain growth. This results in grain refinement and presence of second phase particles also makes dislocation movement more difficult. Second phase particles like chromium carbide in the matrix increases the energy required for elastic/plastic deformation, hence creates higher strength in the alloy.

Percentage of elongation in steels 1, 3 and 4 showed similar results while steel 2 (Ni) with Ni in solid solution showed reduced elongation. Microstructural observation showed presence of acicular ferrite in steel 2. Presence of acicular ferrite has the effects of lowering elongation and increasing yield strength (Kang *et al* 2007). In the case of steel 4 (Ni + Cr), even with Ni and Cr as alloying elements did not show change in ferrite morphology, thus the elongation obtained is equal to the base steel 1. It can be expected that the presence of second phase particles and smaller grain size in steels 3 and 4 should yield less elongation which is contradictory with the experimental results obtained. The explanation can be given from the point of view of the fraction of pearlite present in the microstructure and its distribution. Presence of second phase particle decreased the amount of carbon available in the matrix. Smaller weight fraction of carbon available during cooling should yield lower fraction of pearlite in the microstructure in comparison with the base plain carbon steels 1 and 2 (Ni). Lower volume fraction of pearlite in the microstructure in conjunction with finer distribution increased the deformability in steels 3 and 4. The effects of second phase particles and smaller ferrite grains in decreasing ductility is somewhat countered by the effects of change in distribution and lower fraction of pearlite in the microstructure in increasing ductility.



**Figure 6.** Average grain diameter of steels 1–4.



**Figure 7.** Comparison of yield strength and elongation of steels 1–4 cooled at  $120^\circ\text{C}/\text{min}$ .

#### 4. Conclusions

The carburization technique is a reasonably satisfactory method in revealing the prior austenite grain boundaries in low carbon steels containing nickel and chromium. On heating undissolved particles of chromium carbide refined the austenite grain size. In the presence of nickel, chromium carbide is less effective in austenite grain refinement than chromium carbide in absence of nickel at temperature below 975°C and the reverse is true above 975°C. Nickel did not produce any austenite grain refinement. Nickel and chromium as chromium carbide precipitates were found to refine the ferrite grain size. Cr is found to be more effective in the refinement of ferrite grain size than nickel. Nickel in solution and chromium as chromium carbide precipitates increased the yield strength of the low carbon steels but the effectivity of chromium carbide precipitates in the increment of yield strength was found to be more than that of nickel. In the presence of nickel the contribution of chromium carbide

to increase yield strength is more than that of chromium carbide in the absence of nickel.

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