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Microstructural Evolution and Mechanical Properties Co-relation of Cold-Rolled Ferritic Lightweight Steel with Increasing Carbon

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ABSTRACT

The structure-properties relationship of cold-rolled and annealed Fe-7 wt. % Al lightweight steels for varying carbon contents is explored in this work. Unlike Fe-Mn-Al-C based steels, which experienced processing issues, the hot-worked plates of the present steel were successfully cold-rolled to 2mm thick sheets. Various phases present in the steel for different carbon contents as predicted using the Thermo Calc programme are in line with the experimental findings. The basic alloy with 0.01C contains only a ferrite phase, however, the alloys with higher carbon content have a significant quantity of κ-carbide precipitates. The addition of carbon to Fe-7 wt. % Al steel has improved its tensile strength significantly (438 to 828 MPa). Tensile elongation, on the other hand, has decreased dramatically (26 to 12 per cent) with increasing carbon content The reduction in ductility with increasing carbon is mainly ascribed to the increasing κ-carbide precipitates volume fraction with higher hardness, but not due to the environmental embrittlement as observed in case of higher Al containing steels.

Keywords: Lightweight steel; Low-density steel; Ferritic steel; Iron aluminium alloys; ThermoCalc; Tensile properties

1. INTRODUCTION

Ferritic low-density/lightweight steels are made up of Fe (6-9)wt. per cent Al alloys. These newly discovered steels might be used in lightweight ground transportation systems for military applications such as troop transports, vehicles and armour[1-6]. It might also be used to lighten massive constructions like tunnels and bridges. The mechanical and technological features of these newly produced steels are appealing. They have good ductility and formability, as well as a low specific weight[1,3,5-7]. As a result, these steels offer an intriguing prospect for lightening structures and bridges. Ground transportation systems, such as big trucks and power trains, have other potential applications[1-4].

For the past three decades, substantial research has been conducted in the development of Fe_3Al based intermetallic[8-11] as well as B2 structural alloys[12-13]. These alloys exhibit poor room temperature ductility, toughness, and workability[10-12]. Despite their outstanding oxidation resistance and high strength to weight ratio[8-12]. One strategy for increasing the ductility of these steels has been to reduce the Al concentration to lower values (9wt %) (All composition in wt.%)[6,14]. The reduction in Al content from 9 to 6 wt. percent has been observed to improve room temperature ductility[16,14-17]. This increase in ductility has been ascribed to (i) a lower sensitivity to environmental embrittlement[14-19] and (ii) a decrease in short range ordering from 9 to 6 wt % Al[15,17]. The amount of information available on the manufacturing of lightweight steels (Fe-6-9 wt % Al) based ferritic steel is quite limited[3-7,19]. Various steel firms, such as Arcelor Mittal[20], Usinor[21], and Nippon Steel[22] have demonstrated an increasing interest in these materials, as seen by the number of patents they have published. Furthermore, research on lightweight steels has stimulated the interest of numerous research laboratories and academic institutions throughout the world[1-3,15-16,19,23-24].

Fe-(3-4)Mn-(4-6)Al-(0.1-0.3) C based ferritic steel has recently attracted a lot of attention for the development of lightweight materials for ground transportation systems[25-27]. The tensile strength of these steels is typically in the region of 500-800 MPa, with a ductility of 7-15 per cent. However, it has been claimed that this form of steel cracks during both hot and cold rolling, making it inappropriate for automotive and similar applications.

The development of ferritic lightweight steel for lightweighting solutions has recently yielded promising results[3,14,19,23]. Addition of about 7 wt. % Al to steel has shown promising properties of strength and ductility with reduction in density[6,14-16]. Still, the strength values are lower than the steels used for structural applications[19,23]. Lightweight steels containing Al have the advantages of being less expensive, comprising readily available raw materials, and being able to be produced in large quantities[14-15]. However, it is believed that by raising the specific strength without sacrificing ductility or formability, the field of applications could be expanded to include a variety of new sectors. This is envisaged to be accomplished by alloying steel with carbon to increase its strength[2,4,29]. The effect of hot working on Fe-Al steels with various carbon levels has already been investigated and reported[14-15]. Most of the automobile and other applications

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require these steels in sheet form produced by cold working. However, the studies investigating the carbon addition effect on microstructure, tensile properties and cold workability of lightweight ferritic steel based on Fe-7Al have not been reported. Therefore, a detailed study has been undertaken to identify the optimum carbon content for the best combination of strength and ductility of these steels and the results are reported in this paper.

2. EXPERIMENTAL PROCEDURE

The preparation of raw material and melting technique have been reported previously[2,15]. The procedure to produce plates of 10 mm thickness from 50 mm diameter ingots by hot working process is described elsewhere[14-15]. The hot-rolled plates were cleaned, pickled and were further cold-rolled to sheets of 2mm thickness without any intermediate annealing in two high rolling mill. The 2 mm thick cold-rolled sheets of steels with upto 0.5 wt. % carbon were annealed at 1023 K for 1 hr and then characterized.

Using ThermoCalc software's TCFE6 database, thermodynamic calculations were done to identify phases present at various temperatures and to examine phase changes in steels. The cold-rolled alloy sheets density was determined by Archemedes principle using a Sartorius density measuring kit The mean of five independent readings was taken. Cold-rolled and annealed sheets with longitudinal sections of 2 mm thickness were cut and manually polished to a 0.5-µm finish. The 3.3 ml HNO_3 +3.3 ml CH_3COOH +90 ml H2O +1 ml HF etchant was used for microstructural investigation utilising both optical and Scanning Electron Microscopy (SEM). The phases contained in these steels were identified using a Philips X-ray diffractometer with monochromatic CuK (=1.540562) radiation. The crystal structure of each individual phase was determined by comparing the distinctive X-ray diffraction (XRD) peaks to JCPDS data.

The Vickers hardness machine with a 30 kg load was used to test the bulk hardness of the identical materials. The phases' microhardness was measured applying 10 g load in a Leitz Microhardness testing machine on the etched microscope samples. An average of five measurements was taken to report each hardness data point. Tensile specimens of 4.0 mm width and 20 mm length in longitudinal direction according to ASTM-E 8M for room temperature were machined and polished using 600 grit from 2 mm thick cold-rolled and annealed sheets. Universal testing machine of 'Instron 5500R' make was used to carry out the tensile tests at strain rate of 8.3 x $10^{-4}s^{-1}$. Each tensile test data reported here represents an average of three measurements. Fractography was carried out on tensile tested specimens using SEM.

3. RESULTS AND DISCUSSION

The hot-rolled plates containing up to 0.5 wt. % C could be successfully cold-rolled to 2mm thick sheets. The density of cold-rolled and annealed steel with different carbon content is given in Table 1. As the carbon content is increased from 0.01 to 0.5wt.%, a reduction in the density is observed in the steels from 7247.8 to 7186.2 kg/m^3. These lightweight steels with different carbon content show a considerable reduction in density (~ 10 %) compared to the traditional steel (7873.2 kg/m^3). This is one of the important aspects for the development of structural steels particularly for the ground transport system, where light weighting of the components is an ever-increasing demand[1,3,7].

Table 1. Density of cold-rolled and annealed Fe-7Al light weight steels with different carbon content

Nominal Steel Composition (wt.%)	Density (kg/m^3)
Fe-7Al-0.01C	7247±14
Fe-7Al-0.15C	7226±11
Fe-7Al-0.35C	7212±9
Fe-7Al-0.50C	7186±8

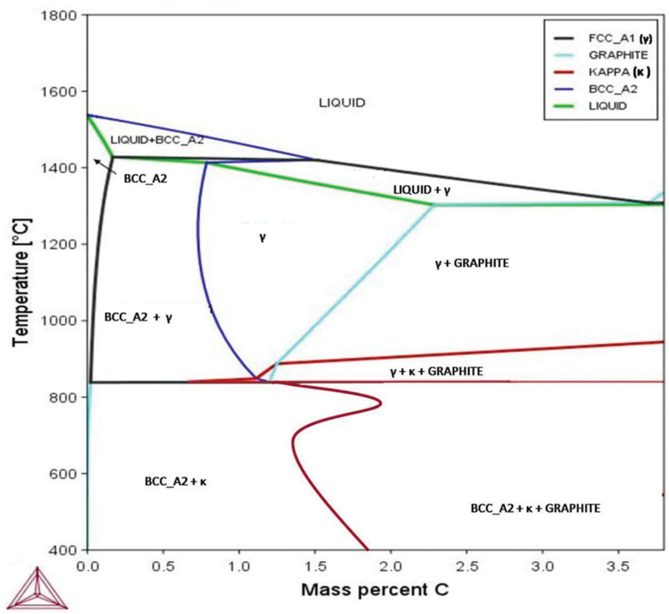

Figure 1. Equilibrium phase diagram of Fe-Al-C with fixed Al (7 %) and varying carbon content as predicted using ThermoCalc.

The predicted equilibrium phase diagram of Al (7 wt. %) containing steel with varying carbon content using ThermoCalc software is displayed in Fig. 1. As the carbon content increases from 0.01 to 0.5 wt. %, the solidification start-temperature of the steel decreases from 1536 ºC to 1505 ºC. The κ-carbide ($Fe_3AlC_{0.5}$) forms at about 810ºC. There is no presence of austenite phase in the steel with 0.01C. At higher carbon content, the austenite phase is observed between 812 ºC to 1426 ºC. The various phases present in these steels with 0.01 to 0.50 wt. % C at room temperatures as predicted by using thermodynamic database are given in Table 2. In case of steel with 0.01C, the stable phases at room temperature are ferrite and traces of κ-carbide. On the other hand, appreciable amount of κ-carbide in ferrite phase is found in all the other steels (Table 2). The left-out Al in the matrix of these steels is calculated based on the procedure mentioned by Khaple, *et. el.* 2015[4].

The optical micrographs of cold-rolled and annealed sheets of Fe-7Al steel containing 0.01 to 0.5 wt % carbon are shown in Fig. 2. The optical micrograph of steel containing 0.01 wt. % carbon exhibited single phase microstructure.

Table 2. Volume fraction of phases calculated for cold-rolled and annealed Fe-7Al steels with different carbon content

Nominal steel composition (wt.%)	Volume fraction of phases (%)			Al left out in the matrix wt.%)
	Predicted by ThermoCalc $Fe_3AlC_{0.5}$ (κ-carbide) Precipitate	Measured using image analyser		Al in Fe-Al matrix
		$Fe_3AlC_{0.5}$ (κ-carbide) precipitate	Ferrite matrix	
Fe-7Al-0.01C	00.45	0.73	99.55	6.95
Fe-7Al-0.15C	05.47	6.68	94.53	6.33
Fe-7Al-0.35C	12.14	12.84	89.11	5.65
Fe-7Al-0.50C	18.02	18.14	81.90	4.75

Calculation procedure for the leftover Al in the matrix[4].

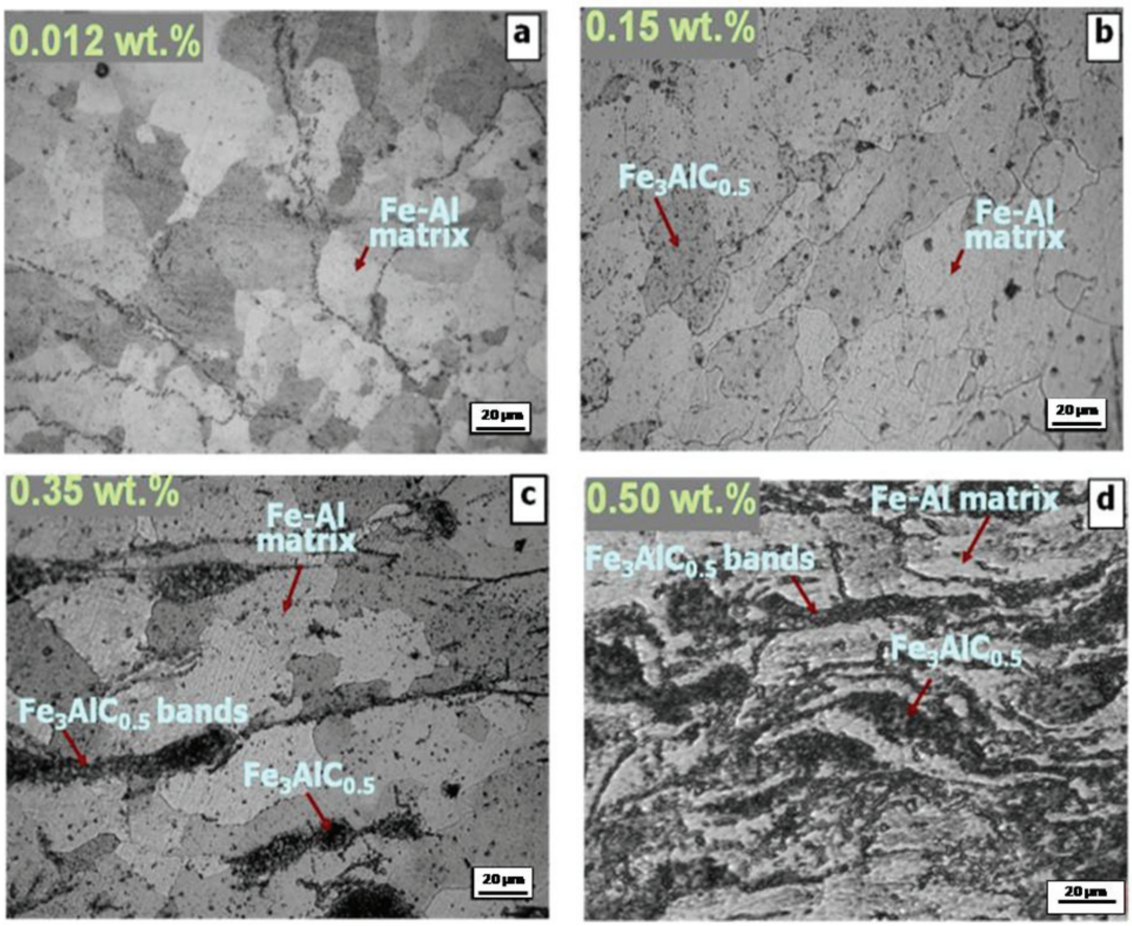

Figure 2. Optical micrographs showing the microstructure of cold-rolled and annealed Fe-7Al steel with (a) 0.01, (b) 0.15, (c) 0.35 and (d) 0.5wt.% carbon.

Steels with greater amounts of carbon (0.15 to 0.5 wt. % C), on the other hand, had a two-phase microstructure with black precipitates in the matrix (Fig. 2b-d). Steel micrographs with 0.35 and 0.5 wt. % carbon reveal a banded structure (in the rolling direction). Dark coloured bands in 0.35 wt. % C steel are thin and their thickness rises as the carbon concentration increases. The steel SEM micrographs are shown in Fig. 3. Dark coloured bands in 0.35 wt. % C steel are thin, and their thickness rises as the carbon concentration increases. The steel SEM micrographs are displayed in Fig. 3. These micrographs show that the band region is mostly composed of very fine spherical precipitates, and the volume fraction of these precipitates increases dramatically as carbon concentration increases.Only the ferrite phase was observed for steel containing 0.01 wt. % C, whereas ferrite and $Fe_3AlC_{0.5}$ precipitates were noticed in steels containing 0.15 to 0.5 wt. % C by the XRD[2,4]. The type of phases present in steel with different carbon contents is in line with the predictions using the ThermoCalc software. Similar phases have been reported by others with the addition of carbon to Fe-Al alloys[30-32].

The bulk hardness and micro-hardness of cold-rolled Fe-7 wt % Al based lightweight steel with different carbon contents is given in Table 3. The hardness of the matrix decreases with increasing carbon content whereas there is no major change in the hardness of carbide precipitates (552–595 HV) (Table 3). Increased carbon levels in steel result in the creation of greater volume fraction of $Fe_3AlC_{0.5}$ precipitates requiring more aluminum.

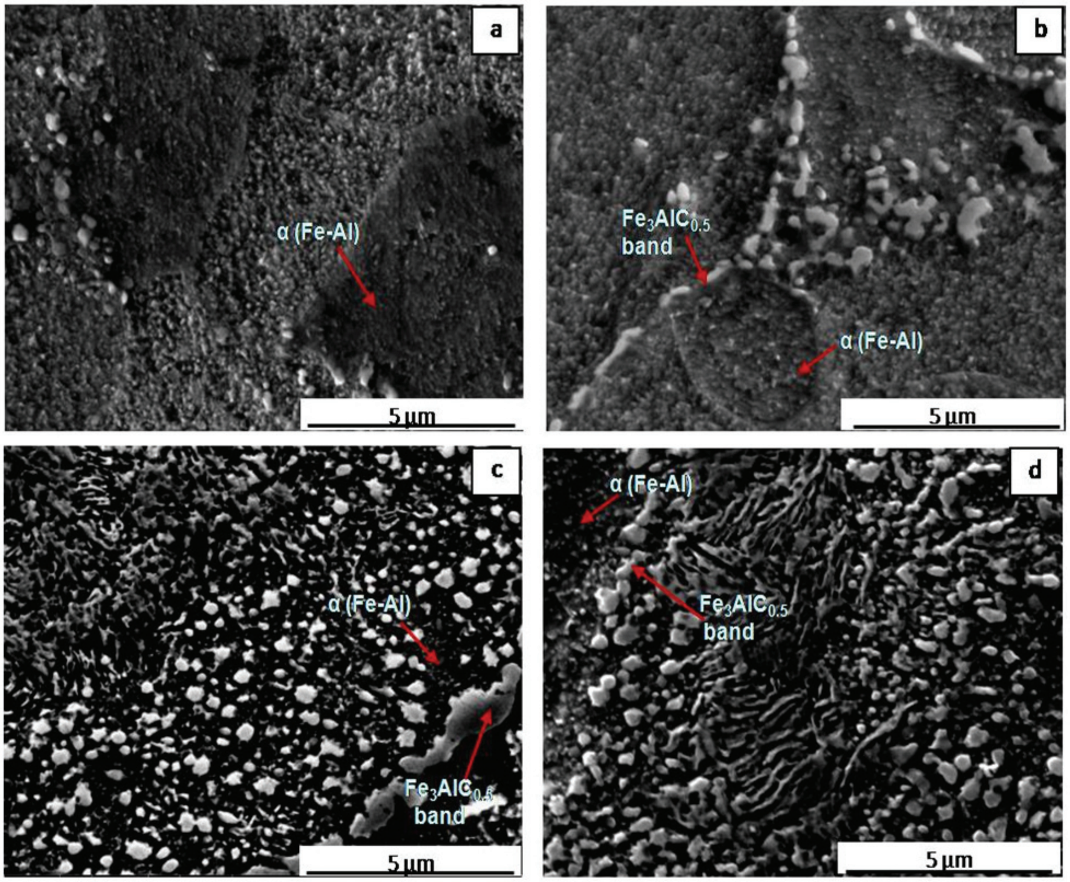

Figure 3. Scanning electron micrographs showing the microstructure of cold-rolled and annealed Fe-7Al steels with (a) 0.01, (b) 0.15, (c) 0.35 and (d) 0.5wt.% carbon. The volume fraction of carbides increases with increase in carbon content.

Table 3. Hardness of cold-rolled and annealed Fe-7Al steels, matrix and carbide with different carbon content

Nominal steel composition wt.%)	Bulk hardness (30 Kg) (H_V)	Micro-hardness (10 gm, Hv) of matrix
Fe-7Al-.012C	198	198
Fe-7Al-0.15C	215	193
Fe-7Al-0.35C	258	182
Fe-7Al-0.50C	298	168

Micro hardness of the carbide precipitates is in the range of (552–595 H_V)

The volume % of the precipitates and the matrix as assessed by the image analyser software is given in Table 2 along with the predicted volume fraction of the precipitates for all the compositions. The volume fraction of the phases reported here is an average of the reading taken at five different locations/frames for each composition. It is clear from Table 2 that as the volume fraction of $Fe_3AlC_{0.5}$phase increases, the concentration of Al in the matrix decreases. This is expected because $Fe_3AlC_{0.5}$precipitates will deplere more aluminum from the matrix.. This results in decrease in hardness of the matrix (Table 3). However, when the carbon concentration(0.01-0.5wt.%) increases, the bulk hardness of the steel increases from 198 to 298 Hv due to increase in volume fraction of carbides (Table 3).

The room temperature tensile stress-strain curves of the cold-rolled and annealed lightweight steel with different carbon content are illustrated in Fig. 4 and the 0.2 % yield strength (YS), ultimate tensile strength (UTS) and percentage elongation % El.) data are presented in Table 4. It can be seen that both yield strength (438 to 828 MPa) and tensile strength (520 to 980 MPa) increase, while ductility decreases considerably with increase in carbon content (from 26 % to 12 %). More than two times increase in the strength is obtained in the steel with the variation of carbon in the above range. It is clear from Table 3,

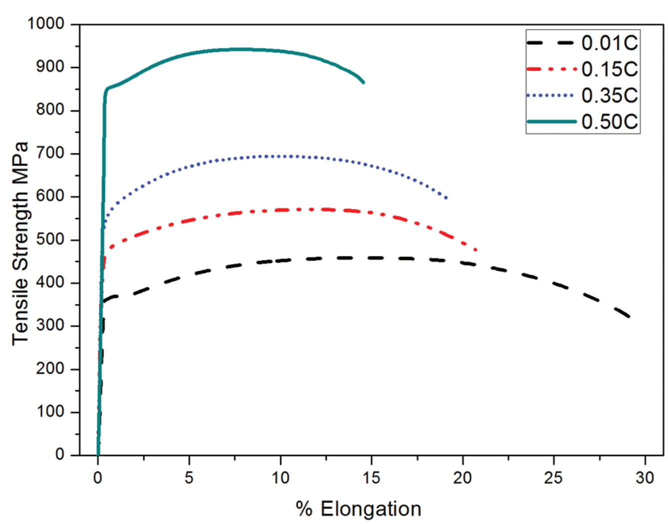

Figure 4. Engineering stress–strain curves for tensile samples of cold-rolled and annealed Fe-7Al steel with different carbon content.

that the hardness of the carbide phase ($Fe_3AlC_{0.5}$) is much higher than that of matrix. Therefore, the increase in hardness as well as yield strength and tensile strength of these lightweight steels with increase in carbon content can be related to the increase in volume percent of carbides. As the size of the carbides is in the range from ~0.1 to ~10 microns, they contribute to the strength of the steel by composite strengthening, i.e., by load transfer mechanism[4,33].

Fe-Al based steels are inherently ductile, however, steel containing higher levels of aluminum (9-16 wt. %) exhibit poor room temperature ductility and toughness. This has been attributed to the environmental embrittlement[15,17-18]. It has been reported that marginal improvement in room temperature ductility[10-11] in high aluminum steels (iron aluminides) can be achieved by the addition of carbon which reduces the susceptibility to environmental embrittlement[10,14-15,18]. In the present study, steels have low concentration of Al (7 %) and hence they are not susceptible to environmental embrittlement. Further, carbon addition leads to the formation of carbides which are found to decrease the ductility. So the decrease in ductility with increase in carbon content may be exclusively attributed to the increase in volume fraction of $Fe_3AlC_{0.5}$ precipitates with high hardness (Table 2).

Table 4. Room temperature tensile properties of cold-rolled and annealed Fe-7Al steels with different carbon content

Nominal steel composition (wt.%)	Ultimate tensile strength (MPa)	Yield strength (MPa)	Elongation (%)
Fe-7Al-0.01C	520±19	438±20	26.0±1
Fe-7Al-0.15C	608±20	510±19	21.3±2
Fe-7Al-0.35C	760±18	652±16	17.1±2
Fe-7Al-0.50C	980±25	828±14	12.4±1

The tensile tested specimens of steels with 0.01 and 0.15 wt % carbon have exhibited higher local deformation near fracture ends than those of steels with 0.35 and 0.5 wt % carbon content. This is in agreement with the higher ductility values of the former than the later steels (Fig. 4). Also, the fracture surfaces of both low carbon containing steel specimens Fig. 5(a) and 5(b) exhibit mixed fracture features of cleavage facets and dimples, with the latter being predominant. On the other hand, the fracture surfaces of both high carbon containing steel specimens (0.35 and 0.5 wt %) exhibit dimples as illustrated in Fig. 5(c) and 5(d). Another interesting observation as can be seen from the magnified images shown in insets of respective

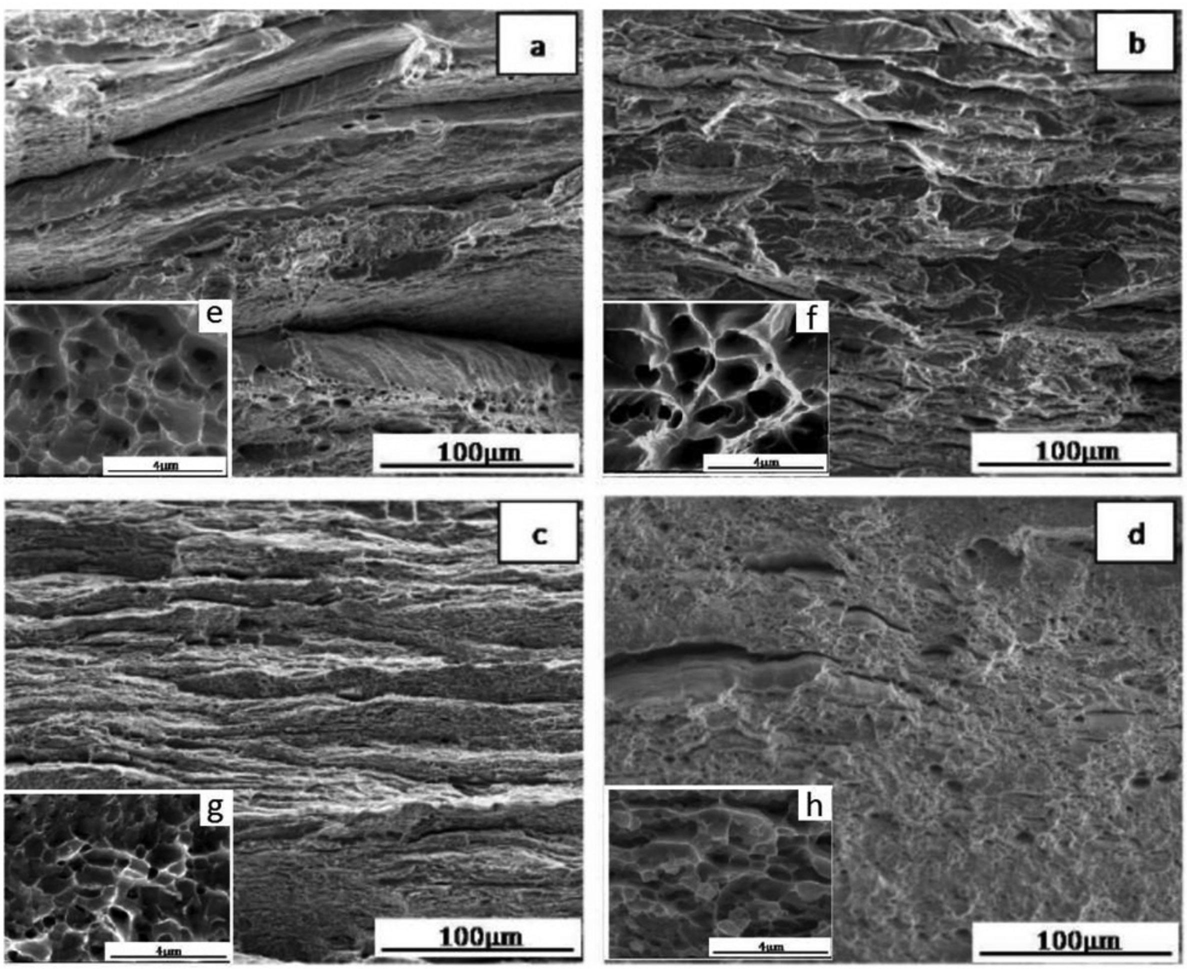

Figure 5. Scanning electron micrographs showing the room temperature tensile fracture surface of cold-rolled and annealed Fe-7Al steel with: (a) 0.01, (b) 0.15, (c) 0.35 and (d) 0.5wt.% carbon. (Insert in the bottom-left are the fracture surface of the same composition at higher magnification respectively).

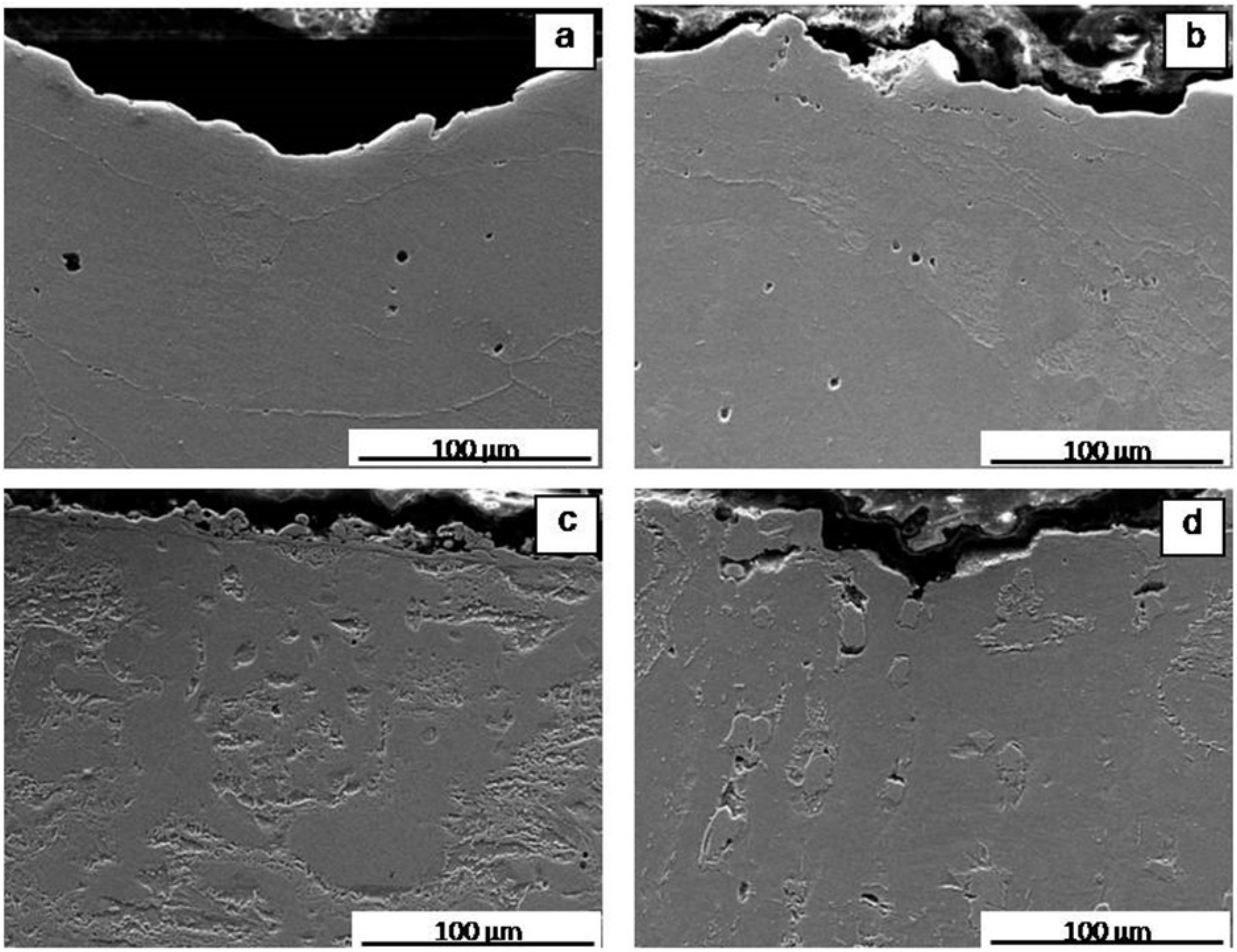

Figure 6. Scanning electron micrographs showing the microstructure of fractured cross-section of cold-rolled and annealed Fe-7Al steel with (a) 0.01, (b) 0.15, (c) 0.35 and (d) 0.5wt.% carbon.

images Fig. 5(a), 5(b), 5(c) and 5(d), is that the dimples are larger but deeper (Fig. 5(a) and 5(b) for steel specimens with lower carbon content (0.01 and 0.15 wt %), whereas the size of the dimples becomes progressively smaller and shallower with increase in carbon content (Fig. 5c and 5d). The dimple fracture features indicate that the fracture mechanism in all tensile steel specimens of different carbon content is essentially by nucleation, growth and coalescence of microvoids formed by decohesion of $Fe_3AlC_{0.5}$ particles from the matrix as shown in Fig. 6. Further, it can also be noted that the volume fraction of $Fe_3AlC_{0.5}$ particles increases with carbon content (Fig. 3 and Table 2) leading to the nucleation of large number of microvoids during tensile deformation. As a consequence, in steel specimens with higher carbon content the number of microvoids are larger thereby the interlinkage of microvoids takes place without much growth leading to fracture of tensile specimen with lower tensile ductility with dimples being smaller and shallower (inset in Fig. 5(d).

In this paper an attempt has been made to compare the tensile properties of various carbon containing lightweight steels with and without manganese (Fig. 7). Lightweight steels containing (0.1 to 0.3 wt. %) Mn show poor ductility (<3%) when tested in the as cold-rolled condition, whereas the same alloys when tested after cold rolling and annealing (973 K/1 hr) have shown good ductility because of the recrystallized grains.

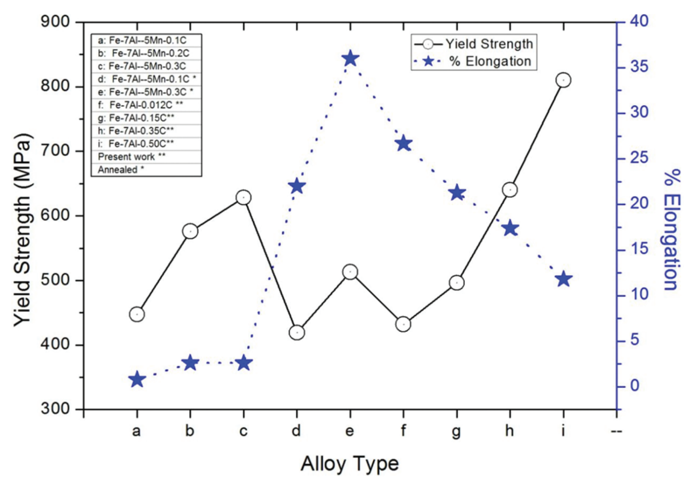

Figure 7. Comparison of yield strength and ductility of carbon containing cold-rolled ferritic lightweight steels with the values from literature[25-27].

Nevertheless these steels are reported to suffer from cracking during hot and cold rolling, even with much lower carbon content (<0.3 wt. %) which may be due to higher hardness of the matrix as well as due to the formation of continuous film of $(Fe,Mn)_3AlC$ carbide[25-27]. However, the present steel (Fig. 7) with 0.35 % carbon and without Mn content have shown good combination of strength and ductility. The tensile ductility is well above 15 % which is considered minimum level for many formability operations like deep drawing, bending etc.

This is considered significant in view of the fact that most of the automobile application require steels in cold-rolled and annealed condition as the final processing step for achieving required tolerances and good surface finish.

4. CONCLUSIONS

The hot-rolled Fe-7Al based lightweight steel (each of 10 mm thickness) with different carbon content was cold-rolled to 2 mm thick sheet. These cold-rolled sheets were annealed and characterized. The workability, hardness and room temperature tensile data is presented.

- The Fe-7Al steel with different carbon levels (0.01-0.5 wt. %) could be successfully cold-rolled. These steels show about 10 % reduction in density compared to conventional steels. This is highly beneficial for automobile and similar applications
- The microstructure of steel with 0.01 wt. % C exhibited a single phase α(Fe-Al) whereas steel with higher carbon exhibited a two-phase structure consisting of $Fe_3AlC_{0.5}$ precipitates in α(Fe-Al) matrix
- The type of the phases and their volume fraction in the steel at different carbon content as predicted using ThermoCalc software agree well with the experimental findings
- Steel with 0.01 wt. % C exhibited excellent (26 % elongation) ductility. However, the addition of higher amount of carbon has resulted in significant reduction in ductility (26 to 12 %)
- The room temperature strength increases significantly (yield strength 438 to 828 MPa, tensile strength 520 to 980 MPa). The enhancement in hardness and strength as well as reduction in ductility with increase in carbon content is solely attributed to the increase in the volume fraction of $Fe_3AlC_{0.5}$ precipitate.

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