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Towards carburizing treatment applied on low alloy steel 16CN6

Benarioua Younes, Maza Samira

Département de Génie Mécanique, Faculté de Technologie, Université de M'sila
BP 166 Ichbilia 28000 M'sila – Algérie
benariouayounes@yahoo.fr

Abstract—**Carburizing technique has recently been developed to engineer the surfaces of the low steels for combined improvement in wear and fatigue resistance. The resultant carburized surface region is characterized by the high saturation of carbon in austenite lattices of steel. The duration and temperature of carburising surface hardening treatment can be chosen in agreement with the thermal treatment for obtaining optimal bulk hardness in the precipitation hardening steel. Characterization point of view structural and mechanical of the samples using X-ray diffraction, optical microscopy and micro indentation testing was then introduced in this work. It was found that the incorporation of carbon resulted in a hardened additional compounds consisting of a combination of martensite and expanded austenite.**

Keywords— **Steel, cementation, carbon, carburizing**

I. Introduction

Carburization is one of the oldest heat treatments used for surface hardening. This process was developed for further improvement of the mechanical properties of the work piece in particular the cutting tools [1-7]. The purpose of this technique is to increase the hardness and wear resistance of the surface by enriching the case with higher rate of carbon (1.2 percent) with subsequent quench hardening; the core of the object, which has not been impregnated with carbon, remains very ductile. Carburising is the addition of carbon to the surface of low-carbon steels at temperatures generally between 850°C and 950°C, at which austenite, with its high solubility for carbon, is the stable crystal structure.

Hardening is accomplished when the high carbon surface layer is quenched to form martensite so that a high-carbon martensitic steel with good wear and fatigue resistance is superimposed on a tough, low-carbon steel core [8-11]. The depth of carburised steel depends of three mainly parameters which are carburising temperature, holding time carburetion treatment and the available carbon potential at the surface [12,13]. When prolonged carburising times are applied for deep depths, a high carbon potential produces a high surface carbon content, which may thus result in excessive retained austenite or free carbides. The work of this study is to obtain cemented steel by pack carburizing treatment. Various time and temperature were used as parameters carburizing treatment.

During this treatment, carbon atoms of powder pack which are present in a great quantity at the surface of low steel diffuse toward the steel substrates in order to combine itself with the iron to form the iron carbide compounds. Effects of carburizing time and temperature on transformation rates of low steel to cemented steel layers were investigated. Micrographs of the samples and thickness of the carburised areas were analyzed by optical microscopy. The structures of different samples were analyzed by X-ray diffraction. Microhardness of cross section of cemented steel samples was measured by using micro Vickers.

II. Materials Experimental Results

A. *Materials and cementation treatment*

The steel (AFNOR 16CN4) was used as treated material in the present work. The chemical composition in weight % is: 0.17%C, 1.22%Cr, 0.88%Ni, 0.46%Mn, 0028%Si, 0.18%Cu, 0.09%Mo, 0.03%Co. The samples to be carburized were machined as cylinders of dimensions 20mm in diameter and 10 mm in length. All specimens were mechanically polished in order to reduce the roughness to Ra = 0.25 µm using sandpaper. Substrates of steels received a pack cementation treatment at various parameters. The carbon was provided by a cement coke powder added with barium carbonate as activator.

The specimens were carburized at different temperature and time of treatment and then were directly quenched in oil. A wide range of processing temperatures between 870° and 930°C and times between 2 and 6 hours have been employed respectively with a pace of 30° and 2h. To provide them with excellent mechanical behavior, all cemented samples underwent a quenching in oil and tempering at a low temperature according to the thermal cycle shown in Figure 1.

B. Characterization and analysis techniques

After pack carburizing treatments carried out at different time and temperature of carburizing, cross-sections were prepared and polished. The samples were then etched in a solution composed of 100 ml of water, 2 ml of HF at 40% and 5 ml of H2O2 at 30%. This type of etching is commonly used to enhance the various zones of cemented steel. Using a B O71 Olympus microscope, this metallographic preparation allowed the observation of morphological details and the thickness of cemented layers. X-ray diffraction analysis was performed to determine the phases at the sample surface using a Phillips diffractometer. The hardness profile of the samples was measured along the cross-sections using a Leco microindenter at lead of 25g.

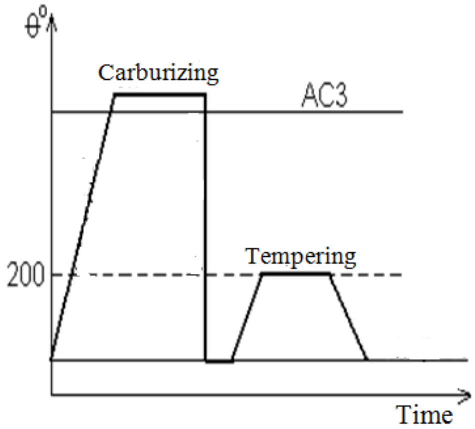

Figure 1: Cycle of carburation treatment used

III. Results and Discussion

Carburizing of the precipitation hardening steels was carried out at various temperatures in order to evaluate the effect of temperature on the resulting microstructure. Additionally, the effect of different time of holding treatment was then investigated. The carburizing potential is directly proportional to the carbon activity and, hence, determines the maximum possible carbon diffused, and governs which phases can be formed.

A. *Morphology*

Micrographs of polished and etched sections of the samples are shown in Fig. 2. Optical microscopy observation show a black zone of cemented steel in surface after carburizing

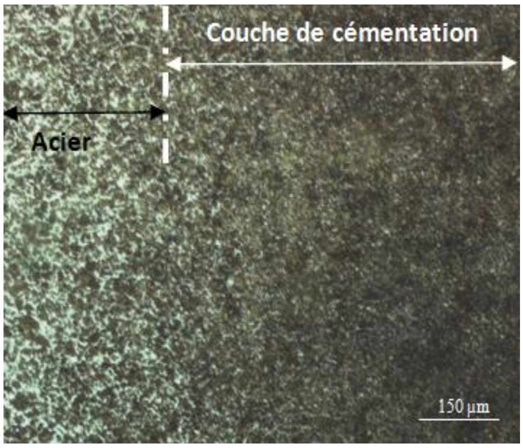

Figure 2: Micrograph of cross section of steel carburized at 930°C for 2h of time

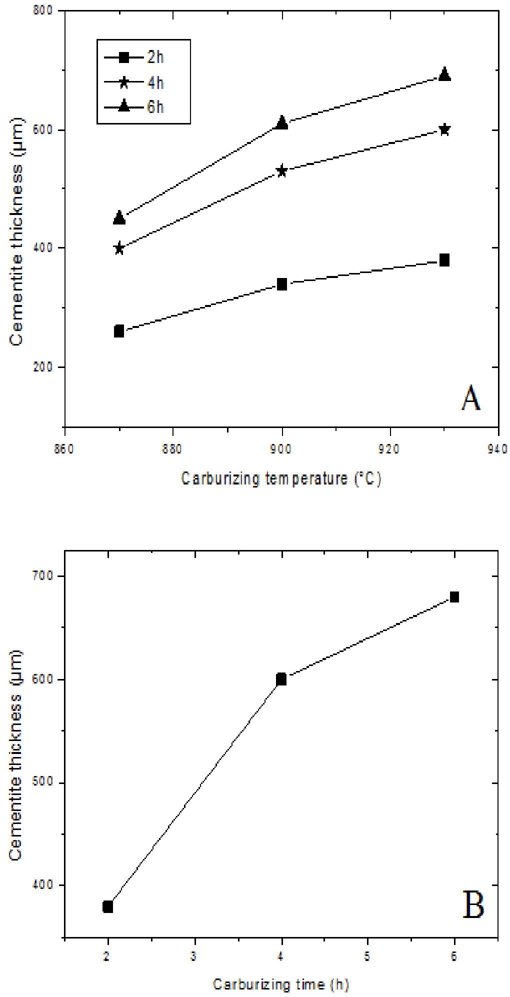

Fig. 3: Variation of carburizing steel in function of A) temperature and B) time thickness of treatment

treatment at 930°C for 2h of time. In this case, the thickness of the cemented steel region was estimated to be 1 mm. This last is subdivided in an outer zone of approximately 0.3 mm and an inner diffusion layer of 1mm. Mainly, carbon atoms have diffused toward the surface and, as a result, new phases involving carbon atoms would be found in the surface area of low steel. The martensitic microstructure is evident in the bulk of the sample.

Processing temperature has the most dominant effect on the structures and the development of the carburized layer. Fig. 3a shows the variation of layer thickness with temperature for the three materials investigated. It can be seen that, in accordance with diffusion-controlled mechanisms, the layer thickness increases with temperature**.** Figure 3b shows the thickness evolution of the cemented steel zone obtained by carburizing treatment at 930 °C for three holding times applied. The thickness of carburizing zone increases with increasing of time and the revealed curve present a parabolic shape.

B. *Structure*

The carburized samples were characterized with X-ray diffraction in order to identify the phases that have developed in the carburized layer. Figure 4 present X-ray diffraction patterns of carburized steel at temperature of 870, 900 and 930°C for 2 h of holding. Fig. 5 depicts the diffraction pattern of steel carburized at 930°C for holding time of 4h and 6h. The X-ray diffraction pattern for steel carburized at 870°C unambiguously shows that several iron carbides phase of Fe_3C, Fe_2C, Fe_3C_2 and $Fe_{2,3}C$ have formed during this treatment.

During carburising at austenitic temperature the steel substrates is converted into carbon rich martensite. Incorporation of diffused carbon produces a distorted structure which causes the precipitation of iron carbides phases. Theses iron carbides phase were restructured when carburizing temperature was increased. In pattern of X-ray diffraction additional peaks are observed which could be ascribed to martensite. Obviously, the carburizing potential plays a major role in determining which phases form during the carburizing process. The peak splitting and the intensity the plan reflections can be explained the prevailing texture of metallic compound formed by carburizing treatment. For carburizing temperature of 930°C, the progression of carburizing transformation increases with the holding time from 2 to 6 h.

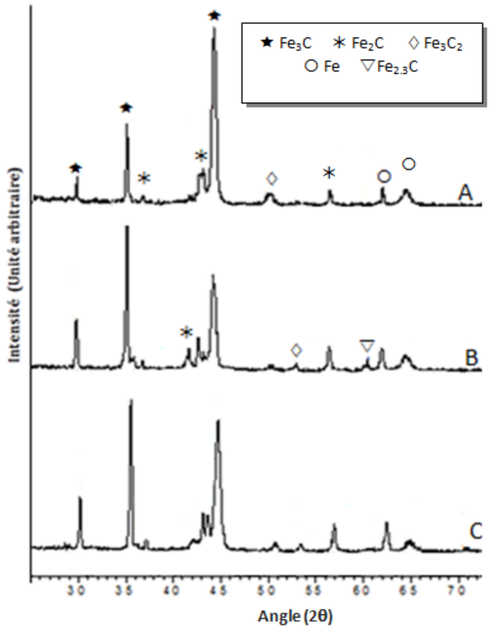

Figure 4: Diffraction spectra of steel carburized for 2h at 870°C (A), 900°C (B), 930°C (C)

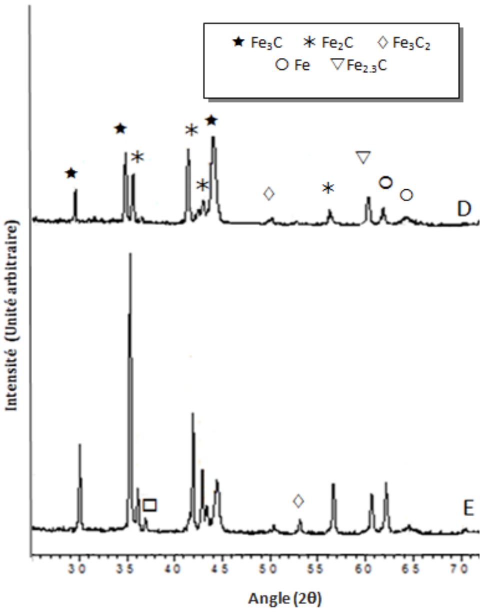

Fig. 5: Diffraction spectra of steel carburized at 930°C for 4h (D), 6h(E),

The X-ray diffraction pattern confirm that precipitation of stable iron carbides begins at low carburizing time and progresses for intermediate holding time, the carbon atoms necessary for the carbide formation coming from the carbon source. The steel treated at carburizing temperature of 930°C for 2h of holding time reveals almost the same structural character then the others carburizing temperatures used in this case. Iron carbide was formed by carbon diffusion from cementation back to low steel at temperature carburizing for 2 h of time. The rate precipitation of carburizing steel phases increases with the tested time from 2h to 6h. This is can be explained by X-ray diffraction peaks and their relative intensity of desired samples. At time of 6h, carburization rate was increased and carburized phases were then textured. Comparing the intensities of samples spectra carburized at different time and temperature as indicated in fig 4 and 5, it is concluded that the steel treated respectively at time and temperature of 6h and 930°C fig.5e, has a dominant texture.

C. *Growth kinetics*

If one considers that the global diffusion / precipitation depth process conducting to the formation of the cemented steel is the same at all carburizing temperature, then the kinetic growth of the produced film should be proportional to the square thickness according to the classical kinetic theory as follow expression (1):

$$d^2 = Kt$$

where d is the thickness of the layer in cm, K is the kinetic growth in cm2 s-1 and t the treating time in s.

Furthermore, the kinetic growth should also be linearly related to the inverse of the carburizing temperature according to the Arrhenius equation (2) associated to thermally activated process.

$$\mathrm{Ln}K = \mathrm{Ln}K_0 - Q/_{RT}$$

where Q is the activation energy in J/mol, T is the tempe rature in Kelvin and R the gas constant equal to 8.31 J/mol K.

fig. 6 shows that relation (2) is satisfied since the experimental points are situated on a same straight line.

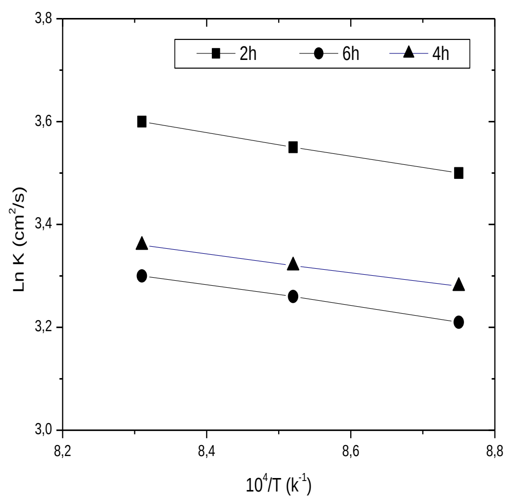

Fig. 6: Growth rate constant versus temperature of carburized steel

The slope of the regression line, lnK in function of 1/T, allows calculating an activation energy of Q = 45.43 kJ mol for various holding carburizing time. Siliva and Mclellan [14] have also measured the diffusion of carbon into bbc iron and found activation energy of Q=18.65 kJ/mol which is low compared to the activation energy required for the cemented steel formation.

In the present work, the iron carbides layers extension should be related to the carbide precipitation process rather than the diffusion process specially if we take into account that the carbon concentration at the surface is not constant but decreases during the heat treatment.

D. *Hardness*

In order to determine the hardness of the carburizing steel in each situation of samples, we perform on their polished sections first Vickers indentation for load of 25g. Figure **7** shows the hardness profiles obtained for samples carburized at temperature of 930 °C for various holding time of 2, 4 and 6h. It is shown that the profile hardness curves present similar forms which have three distinct levels. Interestingly, the locations of these levels are commensurate with the locations of the three region observed with optical microscopy. The first level corresponding to the outer layer present a very high hardness. This last characteristic can be explained by hard compounds of martensitic and new iron carbides produced in this region during the carburizing treatment. In the second level corresponding to the diffusion region, the hardness takes values in descending order which is explained by decreasing the rate of carbon diffused on steel substrates. For the third level, the hardness takes a stationary hardness value which corresponds then to the steel substrate.

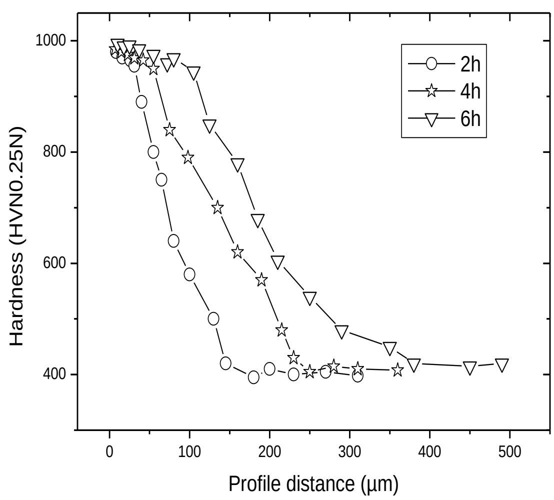

Fig. 7: Hardnes of steel carburized at 930°C for times of 2, 4 and 6h

For carburizing time of 2, 4 and 6h, the hardness of the carburizing steel is almost constant throughout three depth profile corresponding to their samples. The characterization allows for confirming the thickness of three levels of cemented steel in each sample observed optically. The respective mean values of **25** and 60 and 100 µm of cemented steel thickness (first level) for the specimens treated at holding time of 2, 4 and 6h are close to those obtained using metallographic analysis.

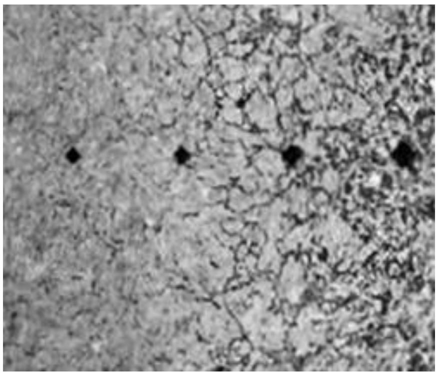

Fig. 8: Micrograph of hardness impressions of carburized steel (930°C-2h),

From the hardness impressions in Fig. 8, it is also evident that the hardness of the carburized steel is significantly higher than that of the substrate. The hardness of the layer decreases gradually from the surface towards the layer core interface, resulting in a diffuse-type hardness profile similar in shape to the hardness profiles shown in Fig. 7.

IV. Conclusions

After carburizing treatments performed on low substrates at various time and temperature, the following points were demonstrated:
1) It is possible to transform the low steel into cementite steel by using only carbon atoms coming from the powder of carburization pack ntoured cemented zone of steel substrate.
2) It is possible to optimize temperature and time of treatment since it was shown that for 6 h of treatment the surface steel was transformed on cemented steel at 930°C.
3) Hardness of layers produced by carburizing treatment was increased even only with a transformation of surface area of low steel into cemented steel. It was found that the incorporation of carbon resulted in a hardened case consisting of a combination of martensite and expanded austenite

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