

Theoretical and experimental treatment of gaseous cementation of iron

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Abstract: Mathematical model is developed for cementation of iron taking into account the diffusion of atomic carbon C through the γ phase. Analytical solutions are obtained assuming constant diffusion coefficients, firstly the analytical method proposed that test to control the process of gaseous cementation, controlled the technological parameters of the cementation such: time (t), temperature (T), initial concentration (C_0), potential carbon or atmospheric concentration (C_{atm}), and speed of the gas flow (xw), secondly to accelerate the process of the gaseous cementation. Finally the results are quantitatively compared with those obtained experimentally taking into account the micro hardness profile. In addition, it is shown that the layer cemented produced during cementation of iron can be predicted by the numerical simulation.

Keywords: Gaseous Cementation, Iron, Ageing Time, Phase Diagram FeC

1. Introduction

The cementation is a thermo chemicals surface treatments by which the atomic carbon C is introduced into steel work pieces content to carbon between 0.10% to 0.18%C, usually at 850°C 1050°C in an endothermic furnace having for object the enrichment in carbon of a superficial layer, of the piece to treat. This enrichment makes himself by stake in contact with a middle rich on carbon solid, liquid or gas. The process the more utilised is the one of gaseous cementation, that is adjusts in series to the industrial productions. The temperature of cementation was maintained the most constant possible T (850-1050) °C and it in a triple but (to avoid the too fast impoverishment of the cement, to facilitate the regeneration avoiding the stay at the high temperature and to decrease risks of piece distortion). Because the solubility of the carbon in phase austenite, being much order elevated than in phase ferrite, and the coefficient of diffusion increase with the temperature, the cementation is achieved to temperatures superior to the A3 line, of the phase diagram FeC, these temperatures lets stable the austenite, solvent in quantity the carbon [1]. In the metallographic study of carburized steel, the structure of the high-carbon case is usually very important. After high-temperature

exposure in a carbonaceous environment, the carburized steel is then hardened, either by direct quenching from the austenitizing temperature during carburization or by reheating and quenching. The amount and morphology of martensite in this case depends on the carbon content and the transformation kinetics. Other microstructural constituents *ie* (retained austenite, nonmartensitic transformation products, carbides and inclusions) also may be present and influence properties and performance. The depth and amount of carbon diffusion depends on the source of surface carbon and the processing time at temperature. The most common method is gas carburization in an endothermic furnace at temperatures in the range of approximately 850 to 1050 °C. The higher processing temperatures of vacuum or plasma carburization (approximately 1050 °C) also allow higher diffusion rates and the additional benefit of increased solubility of carbon in austenite at a higher temperature. At a higher temperature, the surface of the hot steel part can become saturated to a higher carbon content, which thus increases the diffusivity (and hence the carburizing rate). Once the sample is carburized, the hardening step involves rapid quenching from austenite [2,3]. The depth of the martensitic layer depends of the amount of carbon that can diffuse into the sample.

This depends on the carbon content at the surface and the time-temperature exposure during carburization. Sometimes one adopts that the technical thickness of the layer is the one of which the structure after tempering decomposes in martensite (95%) + retained austenite, sometimes one calls him efficient thickness, who corresponds to the depth for which the HRC toughness is superior to 55HRC after tempering to low temperature [1]. The thickness of the layer cemented of pieces made of steel is lower or equal to 17% of the thickness total, which vary between 0.5 and 2 mm. In the metallographic study of carburized steel, the structure of the high-carbon case is usually very important. Moderate amounts of retained austenite are proper and unavoidable in the high-carbon case microstructure of carburized steels. However, excessive amounts of retained austenite can lower hardness to unacceptable levels. Excess retained austenite also can lead to grinding problems. The most important cause of excessive amounts of retained austenite is due to the high carbon on the surface. This condition leads martensite start (Ms) temperatures down, causes the formation of plate martensite, and can shift the balance of the temperature range for martensite transformation to well below room temperature. High alloy content also lowers Ms temperatures. The depth and the amount of carbon diffused depend on the carbon source and the processing time at the temperature T.

Common locations of excessive surface carbon concentration are specimen corners at which the austenite is saturated with carbon during the first part of a carburizing cycle. Carbon potential control and quenching methods are means of controlling the austenite content. If retained austenite is unacceptably high, re-quenching from a lower temperature (distortion) may be considered. Retained austenite also can be reduced by low-temperature treatment in order to allow for more transformation due to the lower Ms caused by high carbon content.

2. The Model

2.1. Cementation

The quantity of the substance distributed (q) that crosses an unit section of the transverse surface by unit of time is proportional to the gradient of the concentration (first Fick's law) [4-7]:

$$q = -D \partial C / \partial x$$

Where: C: concentration of the distributing substance, x: coordinate, D: coefficient of proportionality (coefficient of diffusion). The difference between the incoming flux and the retiring flux in the volume 1.dx is equal to the speed of atom accumulation in this volume, therefore we have:

$$\partial C / \partial t = \partial q / \partial x = \partial (D \partial C / \partial x) / \partial x$$

and since D doesn't depend on the concentration C that is given by the Arrhenius's law:

$$D = D_0 \exp(-E / RT)$$

Where D_0 : factor of frequency, E: energy of activation kcal/g.atom, R: constant of gases perfect (2cal/g.atom), T: temperature °C. The equation of diffusion becomes to only one dimension: $dC / dt = D d^2 C / dx^2$, this equation can be resolved with applying initial and to limit conditions as following :

$$C(x, 0) = C_0 \quad x > 0$$

$$-D \partial C(0, t) / \partial x = \beta' (C_{atm} - C_{ini}) \quad t > 0$$

$$dC(\infty, t) / dx = 0$$

$$C(\infty, t) = C_0$$

Where C: concentration of the carbon % (in mass), C_0 : initial concentration of the carbon in steel % (in mass).

C_{atm} : Potential carbon of the atmosphere %C.

t: time in second(s).

D: coefficient of diffusion of the carbon in the austenite (cm^2/s).

x: co-ordinated in sense perpendicular to the surface (cm).

$$\beta' = (0.25 + 0.63(\omega - 0.2)^{0.66}) 10^{-6} \text{ g} / \text{cm}^2 \text{ s} \% \text{C}.$$

ω varies between 0.2 and 3.2 m/s. In the practice of the gaseous cementation of the steel, the speed of the gaseous flux is usually between 0.3 to 1.5 m/s. The solution of the unidirectional equation differential is:

$$C(x, t) = C_0 \text{Erfc}(x / (2\sqrt{Dt}))$$

$$\text{Erfc}(x) = 1 - \text{Erf}(x) = 1 - 2 / (\sqrt{\pi}) \int_0^x \exp(-u^2) du \quad (1)$$

We used several analytic methods to elaborate this paper:

- Gauss's method, which is the general case for the resolution of the equation of heat propagation [8].

- Taylor's method of development, who assures the partial derivatives of the function C(x, t) up to the order (n+1).

- Korn's method of development: When the speed of gas supplying is limited, the quantity of element (dm) coming from the atmosphere toward the surface of the piece is proportional, unlike the concentration of balance C(0, ∞) and the concentration at the moment given C(0, t), at the F surface and at the time dt [7]:

$$dm = \beta' (C(0, \infty) - C(0, t)) \cdot F \cdot dt$$

Where (β' : proportionality coefficient, which characterizes the speed of the element surrounding the piece toward the surface). If m is expressed in gram, F in cm^2 , t in second and C in gram by cm^2 , the β' unit is cm/s . If the carbon concentration doesn't exceed the limit of its solubility in the austenite, and the speed of the absorption

is not infinite, the change in concentration in the steel is given by the following equation:

$$\theta(x,t) = \frac{C(x,t) - C_{init}}{C_{atm} - C_{init}} \operatorname{Erfc}(x / (2\sqrt{Dt})) - \exp(\alpha^2 Dt + \alpha x) \cdot \operatorname{Erfc}((x / 2\sqrt{Dt}) + (\alpha\sqrt{Dt}))$$

Where $\alpha = \beta' / D \text{ cm}^{-1}$: relative coefficient of transfer. We can to represent this function under another form:

$$\theta(x,t) = \operatorname{Erfc}(B_i / 2T_i) - \exp(T_i^2 + B_i) \cdot \operatorname{Erfc}(B_i / (2T_i) + T_i) \text{ Where } B_i = \beta' x / D \text{ and } T_i = \beta' \sqrt{Dt} / D$$

On the surface of the metal, i.e. in $x=0$, we have [10]:

$$\theta_{surf} = \theta(0,t) = 1 - \exp(T_i^2) \cdot \operatorname{Erfc}(T_i)$$

The coefficient β' characterize the speed of the interaction of the atmosphere with metal. To facilitate the programming and have values discrete, we use the formula of Korn, who serves to the passage of a continuous sum (integration) to a discreet sum (sum), which we can write:

$$2 / (\sqrt{\pi}) \int_0^\mu \exp(-v^2) dv = 1 - (A_1 z + A_2 z^2 + A_3 z^3 + \dots) \exp(-\mu^2) = 1 - \exp(-\mu^2) \sum_{i=1}^n A_i \cdot z^i \quad (2)$$

$i = 1, 2, 3 \dots n$. Where $z = 1 / (1 + A_0 \mu)$

the constants A_i whose equals to : $A_0 = 0.3275911$; $A_1 = 0.254829592$; $A_2 = -0.28449$; $A_3 = 1.42141341$;

$A_4 = -1.453152027$; $A_5 = 1.061405429$. We limiting the value at $n=3$, that to make a precision from calculation of the integral to about 10^{-7} [11]. We indicate for $\phi(\mu)$, the

$$\theta(x,t) = \phi(B_i / 2T_i) \cdot \exp(-(B_i / 2T_i)^2) - \exp(T_i^2 + B_i) \phi((B_i / 2T_i) + T_i) \cdot \exp(-((B_i / 2T_i) + T_i)^2) = (\phi(B_i / 2T_i) - \phi((B_i / 2T_i) + T_i)) \exp(-(B_i / 2T_i)^2) \quad (4)$$

And finally it's the equation that is resolved with computer, on to take the times as a parameter and x as a variable. To substitute the value of $\theta(x,t)$ in the following equation:

$$C_{x,t} = \theta_{x,t} (C_{atm} - C_{ini}) + C_{ini} \quad (5)$$

we obtain the value of the concentration on a defined times and in different depths of the cementation layer.

2.2. Acceleration gaseous cementation

The cementation consists in enriching superficially in carbon a metallic piece. The process of the cementation is used extensively in the modern industry because it permitting:

- to get a big resistance against wear.
- to get a better holding to tiredness etc.....

In general the process takes place between 900°C and 1100°C , during some hours, the speed of the cementation is located between 0.10 and 0.13 mm/h *ie* to get a layer cemented of depth varying between (1 and 1.5) mm, a variable time was necessary between 10 and 12h.

To solve the problem of the gaseous cementation with change of the potential of the middle it is necessary to solve the second unidirectional Fick's law [12-13]:

$$\operatorname{Erfc}(\mu) = \phi(\mu) \cdot \exp(-\mu^2)$$

Where $\phi(\mu) = \sum_{i=1}^n A_i / (1 + A_0 \mu)^i$; $\mu = x / (2\sqrt{Dt})$, A_i : have the numeric constants and $i=1,2,3,\dots,n$

Of another $\operatorname{Erfc}(\mu) = 1 - 2 / (\sqrt{\pi}) \int_0^\mu \exp(-v^2) dv$ are difficult

to integrate, it's why one uses an approximate method, that assures a sufficient precision and approach much of exact value of the integral. This approximate method is demonstrated by the Korn mathematician. The detail of this integral is as follows:

expression from the right under the sign sum of the equation (2), then the equation (1) has the following form:

$$\operatorname{Erf}(\mu) = 1 - (1 - \phi(\mu) \exp(-\mu^2)) = \phi(\mu) \exp(-\mu^2) \quad (3)$$

To substitute (3) in (1), we have:

$$\partial C / \partial T = D \partial^2 C / \partial x^2 \quad (6)$$

or C: concentration of the carbon

t: time of maintain

D: coefficient of diffusion

x: coordinate according to the depth of the layer cemented.

We know that the coefficient of diffusion of the carbon in the austenite during the cementation depends linearly on the concentration of the carbon to a constant temperature, according to formula following:

$$D = (0,04 + 0,08 C) \exp(-Q/RT) \quad (7)$$

And that the flux atomic J distributing in the piece, that is function of the gradient concentration in the layer according to the first Fick's law:

$$J = -D \cdot \operatorname{grad} C \quad (8)$$

And since we interested solely in the diffusion according to only one axis, the formula (8) takes the form:

$$J = -D \partial C / \partial x$$

We taking formulas as a basis (7) and (8), and taking some limit conditions, which depend strongly on the type

of saturation cycle (combined or unique), one can accelerate the process of the cementation. So in this case it is necessary to solve the linear equation to the partial derived of parabolic type, where D is supposed constant equal to $1,1 \cdot 10^{-7} \text{ cm}^2/\text{s}$, at the temperature of cementation 930°C . The combined cycle and the unique cycle are characterized by the presence, respectively of a constant value during the first stage $1,1\%\text{C}$, and $0,8\%\text{C}$ on the surface of the sample, on the other hand during the second stage of the cobined cycle the potential initial is $1,1\%\text{C}$, who change with time. Mathematically these conditions are presented as:

$$C(0) = 0,8\%\text{C} \text{ for } t \geq 0 \text{ (unique cycle)}$$

$$C(0) = 1,1\%\text{C} \text{ for } 0 \leq t \leq t_1 \text{ (combined cycle).}$$

Where t_1 is the time that indicates the end of the first stage of the combined cycle. According to the literature, during the combined cycle, the second stage of the cycle is characterized by the reduction of the potential of carbon, so that the total quantity of carbon that to distribute in metal, during the first stage remains constant, *ie* that the following equation always remained valid:

$$\int_0^\infty C(x, t) dx = Q_0 = \text{cste} \quad \text{for } t \geq t_1$$

The time of treatment between the potential $1,1\%\text{C}$ and $0,8\%\text{C}$ is negligible compared with the total time of the process, metal doesn't undergo a decarbonize during the change of the potential, and the following condition will be confirmed:

$$\partial C(x, t) / \partial x = 0 \text{ in } x=0 \text{ and for } t \geq t_1$$

The interval $0 \leq x < \infty$ indicate that the variable x varies from the surface to an infinite value of the depth. For the numeric realization of an algorithm, as solution of the problem, we use the different limit approximation, for it the domain of integration is supposed fini, *ie* $0 \leq x \leq x_{\text{max}}$ and conditions to limits of the first and the second genre:

$$C(\infty, t) = C_0, \partial C(\infty, t) / \partial x = 0 \text{ become applicable.}$$

The applied data show that the concentration of the carbon, decreases until the initial concentration in steel in

depth. As using a relatively high value of the depth maximal x_{max} , and at x_{max} the concentration will be equal to the one in steel, *ie*:

$$C(x_{\text{max}}) = 0,25\%\text{C} \text{ for } t \geq 0$$

This condition will be verified in the case of the unique and combined cycle. To solve equations of the parabolic type, taking account of the initial conditions, we suppose that the concentration of the carbon is a constant at $t=0$:

$$C(x, 0) = \text{cste} = 0,25\%\text{C}$$

Therefore the formulation of the mathematical model will be like pursuit:

2.3. Mathematical model

We are going to solve the second Fick's law in conditions to limits as following:

For unique cycle:

$$C(x, 0) = 0,25\%\text{C} \text{ for } t=0$$

$$C(0, t) = 0,8\%\text{C} \text{ for } t \geq 0$$

$$C(x_{\text{max}}, t) = 0,25\%\text{C}$$

For combined cycle:

$$C(x, 0) = 0,25\%\text{C} \text{ for } t=0 \text{ and } x > 0$$

$$C(0, t) = 1,1\%\text{C} \text{ for } 0 \leq t \leq t_1$$

$$\partial C(x, t) / \partial x = 0 \text{ in } x=0 \text{ for } t_1 \leq t \leq t_f$$

$$C(x_{\text{max}}, t) = 0,25\%\text{C}$$

3. Experimental Procedures

These materials were prepared in our laboratory by fusion in a device at a high vacuum (10^{-5} Torr) using pure materials. After the melting the ingots have underwent plastic deformation by cold rolling before the homogenization treatment in order to accelerate the structure homogenization kinetics. The homogenization temperature and aging time were chosen from the equilibrium diagrams [14].

Specimens in the form of discs of diameter 20 mm and thickness about 1 cm were prepared

Table 1 : Technological parameters T ($^\circ\text{C}$), C_0 ($\%\text{C}$), C_{atm} ($\%\text{C}$) and x_w (cm/s) of some simulations sample.

	1	2	3	4	5	6	7	8	9	10	11	12	13 1311
T ($^\circ\text{C}$)	900	950	1000	1050	950	950	950	950	950	950	950	950	950
C_0 ($\%\text{C}$)	0.2	0.2	0.2	0.2	0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2
C_{atm} ($\%\text{C}$)	0.9	0.9	0.9	0.9	0.9	0.9	0.8	1.0	1.2	0.9	0.9	0.9	0.9
x_w (cm/s)	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.0	2.0	3.0

from iron (0.10 - 0.15 at. % C; 0.30 - 0.60 at. % Mn; 0.15 - 0.35 at. % Si balance Iron). For microscopic studies specimens were chemically etched with a concentrated solution of Nital at room temperature for 30–90s. Cementation was performed at 1223 K in a gas mixture of H_2 , N_2 , CO , CH_4 and CO_2 . After carburizing, the specimens were moderately cooled.

4. Results and Discussion

We utilise the technological parameters T ($^\circ\text{C}$), C_0 , C_{atm} and x_w of the following table, we obtained several results, for plotting the different graphs:

Using the resulting of calculation to plot curves $C(x, t)$, versus x that takes values varying between zero (that correspond to the surface), and 0.15 cm (depth of cementation supposed sufficient in our type of piece, with a step equal to 0.01 and it for different exposure times, that can take values (1h-9h) to deal the effect of various process parameters technological as temperature; the initial concentration of carbon in iron C_0 %C; the potential carbon C_{atm} %C; and the speed of the flux x_w (cm/s). In Fig 1, we present the calculated carbon concentration profiles in iron, carburized at ($T=950^\circ\text{C}$, $C_0=0.2\%$ C, $C_{atm}=0.90\%$ C, $x_w=1.4\text{ cm/s}$) for different times. It is noted that the concentration $C(x,t)$ decreases at a given time, and reaches the initial carbon concentration. It is indicating that $C(x,t)$ increases at a given depth, for various exposure times. Fig 2, show the effect of T °C on x_{eff} , for different exposure times 2, 5 and 8 h, of carburized iron at $C_0=0.2\%$ C, $C_{atm}=0.90\%$ C and $x_w=1.4\text{ cm/s}$ conditions. It will be shown that x_{eff} increase better at high temperature, for the same exposure times. Fig 3, show the plot of x_{eff} vs initial concentration C_0 for 2, 5 and 8h. It can be clearly seen that x_{eff} increases with

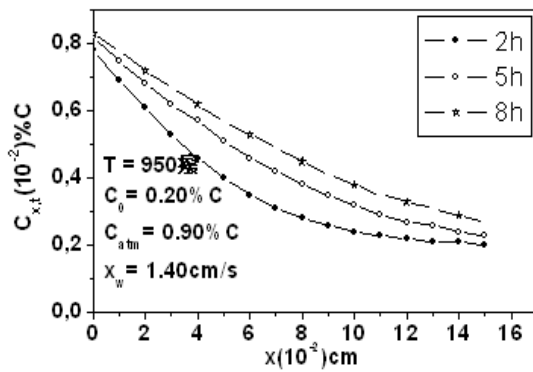


Fig 1. Calculated carbon concentration profiles in iron aging 2, 5 and 8h at 950°C .

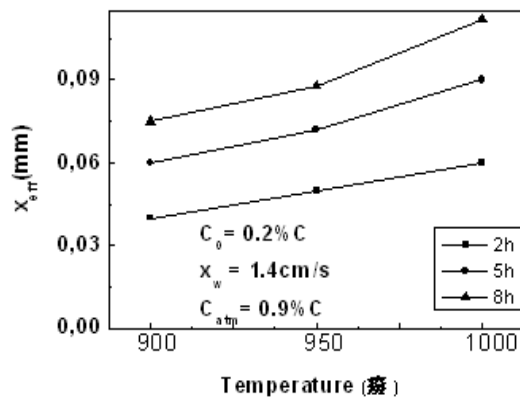


Fig 2. Calculated effect of temperature and ageing time on x_{eff} .

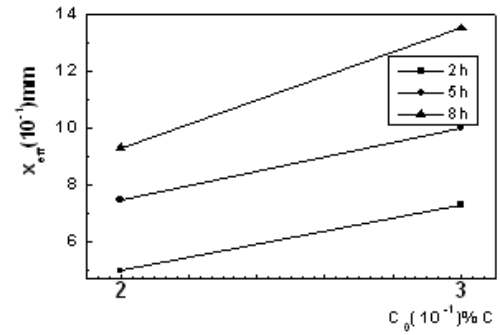


Fig 3. Effect of initial concentration and ageing time on x_{eff} .

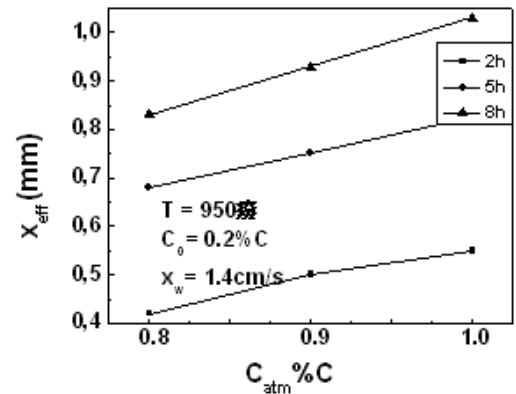


Fig 4. Effect of atmospheric concentration and ageing on x_{eff} .

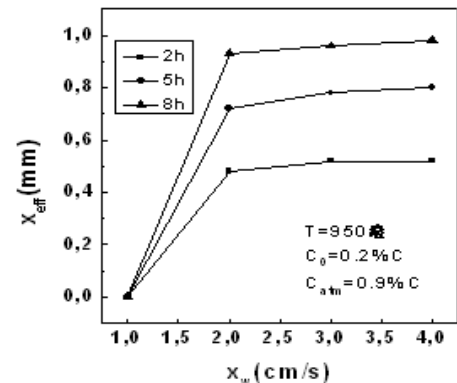


Fig 5. Effects of speed of the gaseous flux and ageing time on x_{eff} .

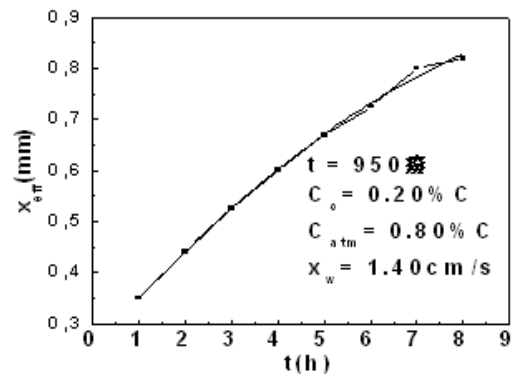


Fig 6. The penetration depth of the carburized layer as a function of ageing time at 950°C .

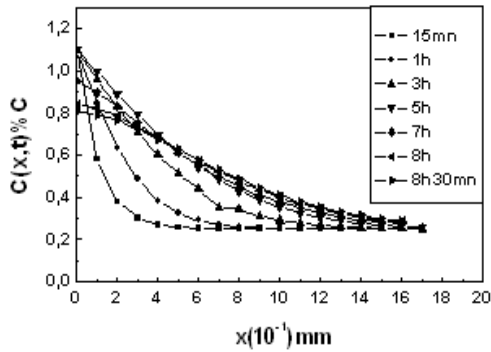


Fig 7. Variation of the carbon concentration according to the depth of the layer cemented, for different ageing time at 950°C.

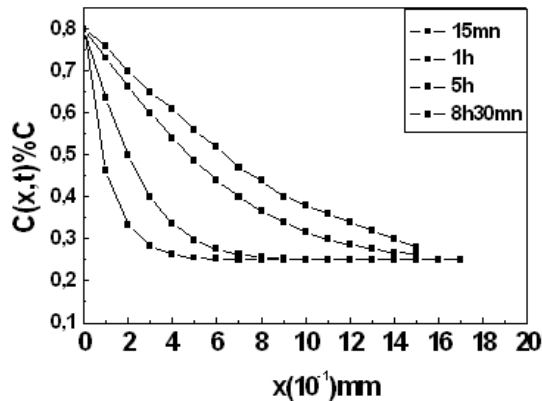


Fig 8. Regime unique: variation of the carbon concentration $C(x, t)$ according to the depth at different time at 950°C.

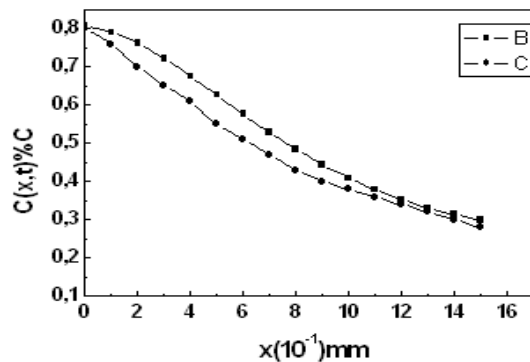


Fig 9. Comparison between curves: diffusion in the unique regime (solid circles) and the combined regime (solid squares).

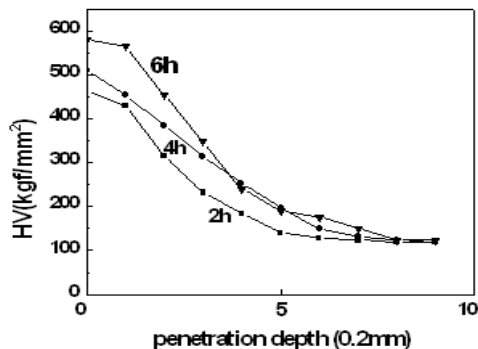


Fig 10. The micro hardness dependence at different ageing time according to the penetration depth.

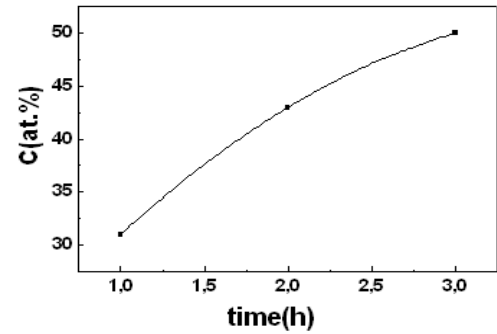


Fig 11. The calculated micro hardness as a function of ageing time at 950°C.

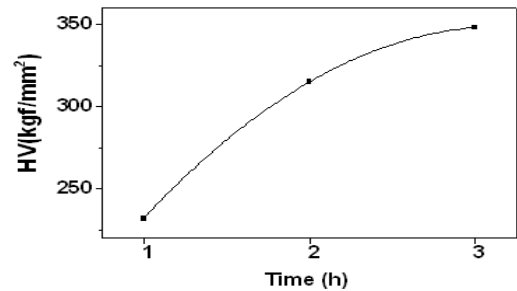


Fig 12. Micro hardness as a function of ageing time at 950°C.

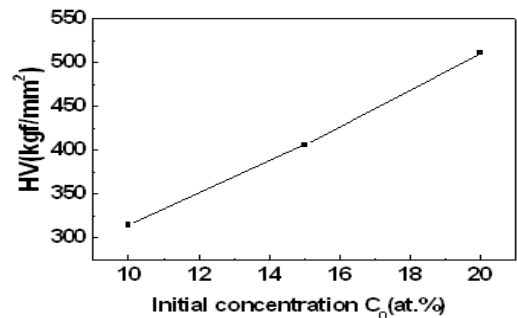


Fig 13. Micro hardness as a function of initial concentration.

Co. It is indicating that x_{eff} increases at a same value of C_0 , for different exposure times. Fig 4, illustrate the dependence of atmospheric content C_{atm} on the x_{eff} . The simulations were performed at $T=950^\circ C$, $C_0=0.2\% C$ and $x_w=1.4\text{ cm/s}$ for various times. It is shown that x_{eff} increases linearly with C_{atm} . It is observed that it increases at a same value of C_{atm} for different times. The plots in Fig (3-4), are seen to be linear, indicating that this reaction is first order. It is apparent that the plots x_{eff} vs C_0 (C_{atm}) increase at the same time and for the same C_0 (C_{atm}) and different exposure time. Fig 5. show the effect of x_w on the sufficient depth for various times, at $T=950^\circ C$, $C_0=0.2\% C$ and $C_{atm}=0.90\% C$. It is apparent that x_{eff} increases quickly for small values of x_w , after it increase slowly and reached the constant values for different exposure time. Fig 6. show the effect of carburization time on x_{eff} , same conditions are in Fig 4, but atmospheric carbon content C_{atm} is equal to $0.8\% C$, that x_{eff} grows parabolically with time and reaches the constant value of potential surface. Fig 7, represents the variation of the carbon concentration according to the depth of the layer cemented, for different ageing time. We notice that: This concentration (for a

stationary depth) increases of one time to another superior. For a concentration fixes, the depth of the layer cemented increases of one time to another superior. Fig 8, represents regime unique: variation of the concentration of the carbon $C(x, t)$ according to the depth of the different ageing time. The concentration decreases in depth since the surface that is equal to 0.8%C, that it reaches the initial steel concentration that is equal to 0.25%C. Fig 9 represents a comparison between curves: diffusion in the unique regime (solid circles) and the combined regime (solid squares). We notices that for a concentration constant, the depth increases with a regime respectively to the other, for the same ageing time. Concentration profiles, as calculated according to the programme, are presented in Fig 1. For a comparison with experiment the micro hardness profiles of the specimens were determined after cementation. The increase in micro hardness with respect to the micro hardness of the non cemented material is given as a function of penetration depth in Fig 10. Fig 11 show the calculated carbon concentration in iron specimens cemented at 950°C, $C_0 = 10\%$ for 2, 4, and 6h. It grows parabolically with time. In Fig 12. we show the increase of micro hardness vs. ageing time. It is clearly seen that an increase of micro hardness corresponds to an increase of carbon concentration, see Fig 11. In Fig 13, we show the increase in micro hardness at $T=950^\circ\text{C}$, $t = 4\text{h}$ and thickness 0.6 mm with initial concentration of carbon in iron specimens, that it is sensitively comparable to Fig 3. A sensitive graphic presentation for comparison of the experimental absorption-isotherm data with the model description of the set of equations (5) is provided by Fig (10-13). So a qualitative agreement exists between the calculated results Fig (1-3, 6). and experimental results.

5. Summary

We have elaborate a mathematical model, a program in data processing of the gaseous cementation, that serves to the survey of the concentration in carbon according to the technological parameters, and of the depth of the layer cemented, $C\% = C\%(x, t, Catm, T, Co, xw)$. While basing on results given by this model, we have study the technological parameters influence on the cemented layer. We deduct some curves presented below that: x_{eff} increases with time (t), with the $T^\circ\text{C}$ temperature, with $Catm$, with Co , and with xw , while taking at every time all constant taken technological parameters. x_{eff} can not increase infinitely because (risk to damage the characteristic mechanical): while increasing the temperature or the time of maintains grains becomes thick (parameter to make fragile). The initial concentration Co is limited (cementation of steels low carbon). We are limited by speed xw that can go until 1.3 cm/s, value taken by the technician like limit.

We know that: the thickness of the layer cemented varies according to the concentration of the carbon according to the equation:

$$C(x, t) = C_0 \text{Erf}(x/(2\sqrt{Dt}))$$

The coefficient of the diffusion D in the austenite increases with the content in carbon to a constant temperature according to the equation $D=0.04+0.06\%C + \dots$

These two phenomena permit to accelerate the gaseous cementation and to get a more preferable carbon distribution (distribution in landing). This method of acceleration is rarely used in the industry, because it is not controlled well.

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