FineCarb® - the flexible system for low pressure carburizing. New options and performance.

Piotr Kula[1], Maciej Korecki[2], Robert Pietrasik[1], Emilia Stańczyk-Wołowiec[1],
Konrad Dybowski[1], Łukasz Kołodziejczyk[1], Radomir Atraszkiewicz[1], Michał Krasowski[1]

[1] *Institute of Materials Science & Engineering, Technical University of Lodz, ul. Stefanowskiego 1, 90-924 Łódź, Poland*
[2] *Seco/Warwick S.A., ul. Sobieskiego 8, 66-200 Świebodzin,, Poland*

The advanced **FineCarb®** technology developed in cooperation between Seco/Warwick S.A and Technical University of Lodz (PL) offers the wide range of classic and less common surface hardening processes for mechanical engineering. The physical model of carbon transfer based on the idea of the intermediate carbon deposit and the "extended boost – diffusion" schedule as combined with the expert system **SimVac Plus™** have been described. Also, the paper presents the **SimHard™** unit that simulates hardness profile after low pressure carburizing followed by high pressure gas quenching. Additionally, the idea of the grain growth limiting by preliminary nitriding **PreNitLPC®** and new methodology for active charge surface control as the new options for **FineCarb®** development are described.

Keywords: *low pressure carburizing, expert system, carbon and hardness profile, grain growth limitation, on-line control.*

1. Physical model of carbon transfer

The complex mixture of gases is applied as an original carburizing atmosphere in **FineCarb®** system. The carbon carrier in a form of preliminary mixed two unsaturated hydrocarbons of specified and patented ratio forms the basis for the carburizing atmosphere [1]. The synergic use of ethylene and acetylene results in advantageous effects and the final outcome of the treatment due to this synergism is enhanced in comparison to atmospheres based on single carbon carriers [1]. Such prepared carbon carrier can be mixed with hydrogen. Consequently, carbon is effectively transferred from a gaseous phase to the charge without any by-product as soot and/or tar creation. The decomposition of hydrocarbons may reach even 70-90% and exhaust gases contain mainly hydrogen.

It has been disclosed that the carbon transfer from hydrocarbon gas mixture to steel proceeds through an intermediate surface phase – "carbon deposit". The deposit consists mainly of: hydrocarbon groups CH_n, represented by methyl group CH_3 (of the reduced mass of the group m/z=15, where m corresponds to the mass of the group and z – the ionization level), methylene CH_2 (m/z=14) and CH (m/z=13), single C atoms (m/z=12), hydrogen atoms (m/z=1) and the groups of atoms Fe-H (m/z=57). The complexes with two carbon atoms are represented by C_2H_3 (m/z=27) and C_2H_5 (m/z=29) groups. The hydrocarbon groups containing three carbon atoms are represented by complexes C_3H_5 (m/z=41) and C_3H_7 (m/z=43). Both the C_1 and C_3 hydrocarbons in the deposits are the proofs that the double C=C bonds of the primary ethylene molecules adsorbed on the iron surface have been broken. The concentration of hydrocarbon complexes containing four and more carbon atoms on the sample surface is very low [2].

The carbon deposit is created during any boost stages and as the mixture of hydrogenated carbon compounds it is a source of active carbon both during boost and initial period of diffusion stages (figure 1).

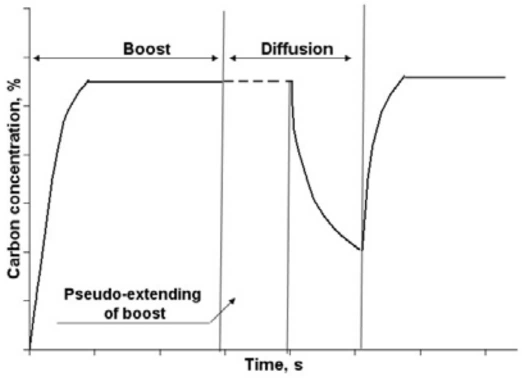


Figure 1 Evolution of a surface carbon concentration during **FineCarb®** low pressure carburizing.

2. SimVac™/SimHard™ expert system

2.1 Simulation of carbon profiles

The described physical model of carbon transfer in the **FineCarb®** is the base for the computational algorithm for the **SimVac™** software. The system offers the computer simulation program for technological vacuum carburizing process structure programming which takes into account a series of factors that influence the final carbon concentration profile and the carburized layer microstructure. It allows programming the optimum structure of the carburizing process and gives consideration to process temperature, grade and chemical composition of the steel, shape (curvature) of the carburized elements and the pre-cooling before quenching procedure. The program bases on the new dependence between diffusion coefficient and temperature and steel chemical composition (correct in the range of the concentrations 0-2,0%), determined experimentally and grounded on realized special, wide schedule of vacuum carburizing.

After the proper parameters are loaded (carburizing temperature and quenching temperature) and the charge parameters (steel grade and characteristics of treated elements curvature) also, user can choose between the manual planning of the process segment structure and automatic one. In the manual option a user, taking his own criteria in following approximations, achieves planned, final carbon concentration profile. Whereas, in automatic option, the segmentation is optimized to obtain, as a result of the process, assumed surface carbon concentration and case depth without exceeding the instantaneous concentration during the whole process, what would result in hardly soluble carbides precipitation. The structure and algorithms of the program guarantee the very quick realization of the calculations both in manual and automatic options.

2.2 Simulation of hardness profiles

The carbon profile that has been obtained by **SimVac™** is next processed by **SimHard™** software to simulate the hardness profile after high pressure gas quenching. To analyze the effectiveness of this hardening, the research group had to address the following design issues [3]:

1. Describe the phenomena accompanying the cooling of the charge inside vacuum carburizing furnace chamber, together with an examination of the model allowing to determine characteristic ξ parameter which defines cooling intensity. It must be mentioned that with regard to the cooling chamber geometry, determination of the $\alpha(t)$ parameter was associated with the requirement for the calculations to include both construction parameters and gas velocity fields inside the chamber [4].
2. Determine the influence of the material grade (chemical composition, physical properties) on the properties obtained after heat treatment. A database was created to tabulate the results.
3. Describe the relationship between the shape, mass and surface area of the charge and cooling intensity.
4. Development of a mathematical model to determine the effect of cooling speed on each point of the charge geometry.

Determining the cooling speed at a particular distance from the surface of analyzed detail was necessary to calculate hardness profile in the carburized layer of a particular geometry. To meet this goal, the group decided to use the superposition method [5, 6, 7]. This solution is based on the transient heat flow equation [8, 9].

To assure the accuracy of the calculations, values λ, ρ and cp were taken as a temperature dependent. Coefficient $\alpha(t)$ in equations determines the intensity of heat exchange between details and the cooling media. The distribution of temperature and gas velocity in the furnace chamber are necessary for heat treatment processes in gases under elevated pressures to determine the heat transfer from the charge to the flowing gas. Fixing these parameters in correlation with known properties of materials such as hardenability, surface and core hardness taking into account changes in carbon content in surface layers of elements after carburizing is the base for accurate simulation of real heat treatment conditions. It was necessary to find the relationship between hardness and cooling speed within a given distance from the surface as well as with carbon concentration [5, 6].

Experimental verification of the results obtained from the described hardness calculations method was performed in a single chamber furnace with vacuum carburizing, type VPT4035/36. The samples tested had dimensions of Ø25x150 and were made of two steel grades: 16MnCr5 and 18CrMnTi5, and underwent different carburizing processes. Each charge contained 270 samples in two layers of total weight 156kg and surface area equal 3,5m^2. Different thickness of case depths with different surface hardness were taken into account to verify calculated by SimVacPlus Hardness program module values.

The SimVacPlus Hardness module was used to calculate carbon profiles and process structure (with division into carburizing and diffusion only stages). Next, the data was imported into the computerized system (Programmable Logic Controller) of the VPT furnace, and the process of vacuum carburizing and quenching in nitrogen under pressure of 9 bars was carried out. Microhardness profiles of treated samples were determined after the processes, with results shown in figures 2 and 3. In case of 16MnCr5 steel varying thickness of the carburized layer, different surface hardness were considered; in case of 18CrMnTi5 steel required surface hardness that was constant, the carburized layer thickness varied.

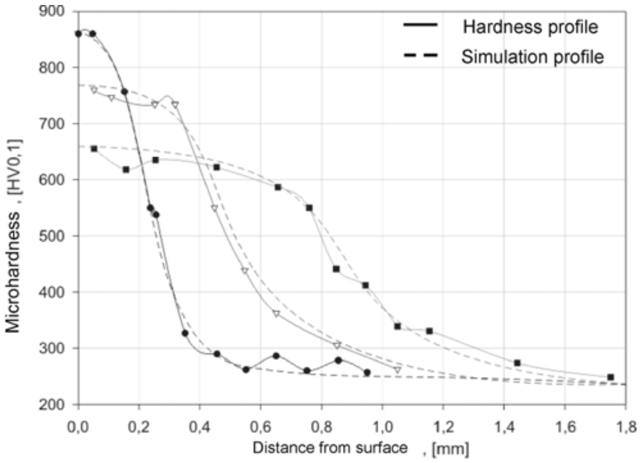


Figure 2. Hardness profiles for 16MnCr5 steel samples (comparison of simulated profiles and experimental data).

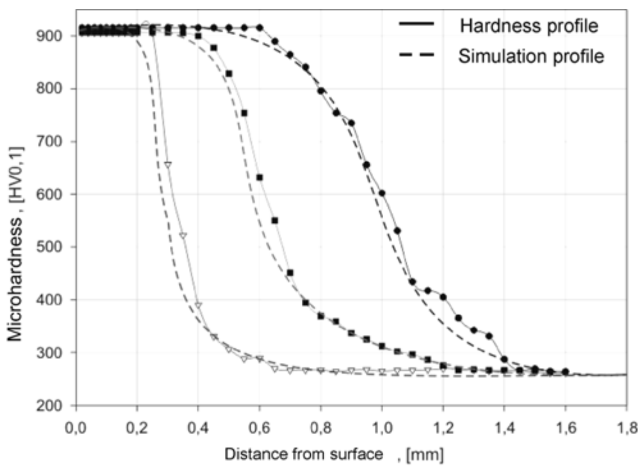
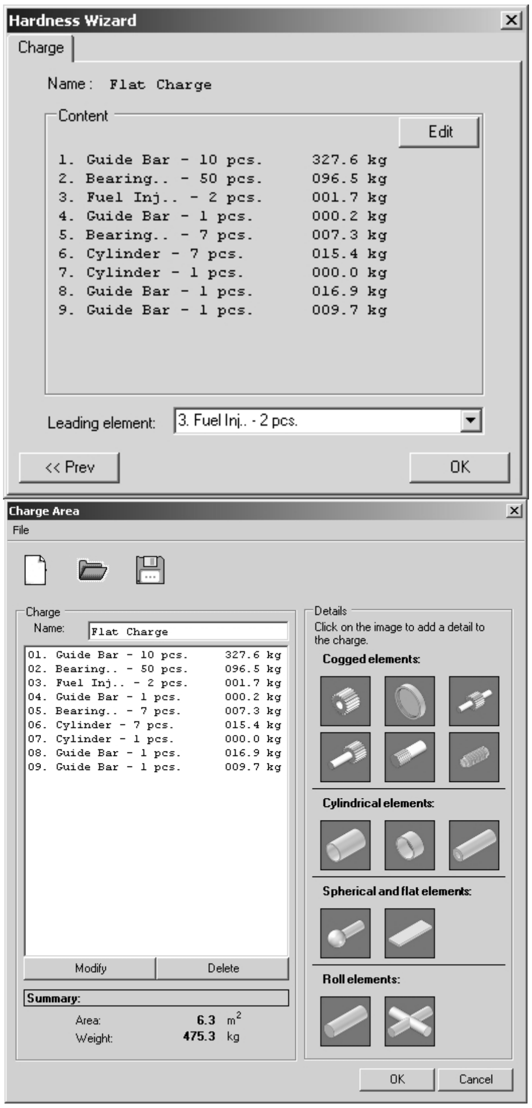


Figure 3. Hardness profiles for 18CrMnTi5 steel samples (comparison of simulated profiles and experimental data).

2.3 Auxiliary options

The critical step, is defining the load to be heat treated. The menu selections were created especially for this purpose, which quickly describe the physical load properties and the surface area to be carburized (figure 4, 5).



Name: Flat Charge

Content

```
1. Guide Bar - 10 pcs.     327.6 kg
2. Bearing.. - 50 pcs.     096.5 kg
3. Fuel Inj.. - 2 pcs.     001.7 kg
4. Guide Bar - 1 pcs.      000.2 kg
5. Bearing.. - 7 pcs.      007.3 kg
6. Cylinder - 7 pcs.       015.4 kg
7. Cylinder - 1 pcs.       000.0 kg
8. Guide Bar - 1 pcs.      016.9 kg
9. Guide Bar - 1 pcs.      009.7 kg
```

Leading element: 3. Fuel Inj.. - 2 pcs.

Name: Flat Charge

```
01. Guide Bar - 10 pcs.     327.6 kg
02. Bearing.. - 50 pcs.     096.5 kg
03. Fuel Inj.. - 2 pcs.     001.7 kg
04. Guide Bar - 1 pcs.      000.2 kg
05. Bearing.. - 7 pcs.      007.3 kg
06. Cylinder - 7 pcs.       015.4 kg
07. Cylinder - 1 pcs.       000.0 kg
08. Guide Bar - 1 pcs.      016.9 kg
09. Guide Bar - 1 pcs.      009.7 kg
```

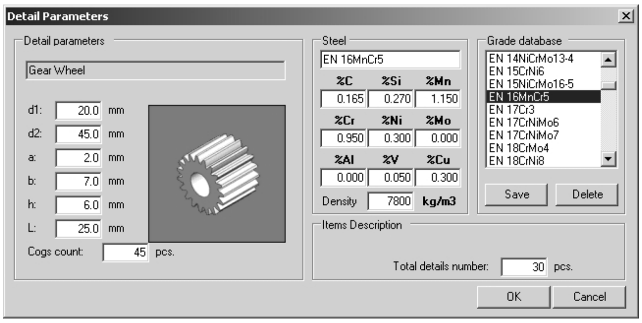
Click on the image to add a detail to the charge.

Summary:

Area: 6.3 m^2

Weight: 475.3 kg

Figure 4. Windows of charge configuration and modifying.



Gear Wheel

d1: 20.0 mm

d2: 45.0 mm

a: 2.0 mm

b: 7.0 mm

h: 6.0 mm

L: 25.0 mm

Cogs count: 45 pcs.

EN 16MnCr5

%C	%Si	%Mn
0.165	0.270	1.150
%Cr	**%Ni**	**%Mo**
0.950	0.300	0.000
%Al	**%V**	**%Cu**
0.000	0.050	0.300

Density 7800 kg/m3

EN 14NiCrMo13-4
EN 15CrNi6
EN 15NiCrMo16-5
EN 16MnCr5
EN 17Cr3
EN 17CrNiMo6
EN 17CrNiMo7
EN 18CrMo4
EN 18CrNi8

Total details number: 30 pcs.

Figure 5. Simulation window for defining charge elements.

Such estimated weight and area of charge are the input data for hardness profile simulation while surface area determines the carburizing gas dosing rate and this parameter is directly transferred to the PLC of vacuum furnace to set mass flow controllers.

3. On-line correction system

The control and monitoring over vacuum carburizing process has significant meaning from practical point of view. However, it is very difficult task because this process is non-equilibrium one.

The most modern monitoring methods apply the concept based on the hydrogen content determination. Two different types of sensors are used. The first one measures the partial pressure of hydrogen [10], while the second is the thermo-conductometric one [11].

All these sensors have been practically used for numerous, reproduced in big-scales elements, i.e. for automotive industry. However, none of these methods are perfect, especially when the details are produced in a small-scale and with constantly changing requirements and parameters of the carburizing surface layer. This is one of the reasons why the vacuum carburizing process is difficult for wide range of applications.

The proper carburizing gas dosing is related to the active charge surface area. The estimation tool for that parameter has been described above. The simple and reliable system for on-line controlling for this setting has been developed for the **FineCarb®** concept. The idea of it is applied herein refers to the measurements of the outlet gases densities. The main elements of this system are shown below.

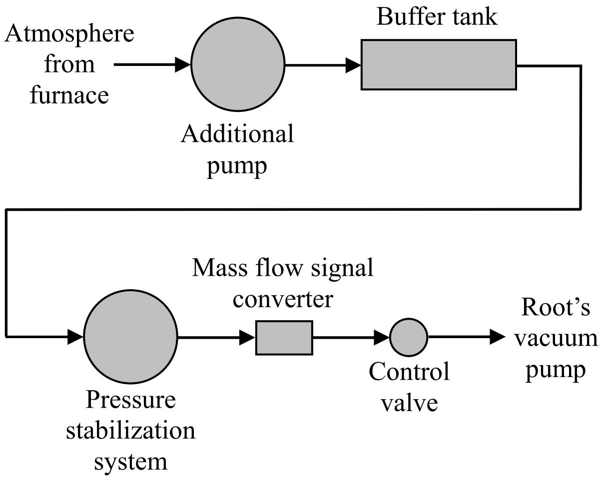


Figure 6. The main components of a on-line control system.

The nature of the method is based on the fact that the signals from the mass flow converter, ones which are collected in the certain time intervals during the first boost phase, are transmitted to the expert system. Next, the average values of outlet gases densities are compared with the experimentally fixed ones in the function of the active charge surface, with model characteristics. The calculation of the correction for the accepted in the system established charge surface is done in the following step and is sent to the process simulator and then to the furnace controller. The next boost phases are done according to the corrected flow, if needed. As a result in the course of the process, one achieves regular carburized layers of a correct shape, layers of carbon concentration complex profile, and avoids the creation of by-products, such as tar and soot [12].

The method and the system, one constituting the compact measurement system, do eliminate the risk of charge damage as well as/or installation damage resulting from the possibility of error and imprecise data on the area of the treated elements input by the operator.

4. PreNitLPC® – fine grain option

The prenitriding option **PreNitLPC®** has been developed for extending of the application range for **FineCarb®** technology towards higher carburizing temperatures and wider range of steel grades. Technically, it is based on the dosing of ammonia gas to the vacuum furnace chamber during continuous heating of charge in the temperature interval from 400°C to the moment the charge reaches the carburizing temperature. The nitrogen saturates delicately the surface layer and protects against the austenite grain growth before the diffusing carbon takes over this function [13]. Then nitrogen is homogenously dissolved in deeper zone (figure 7) and it doesn't cause a retained austenite stabilization.

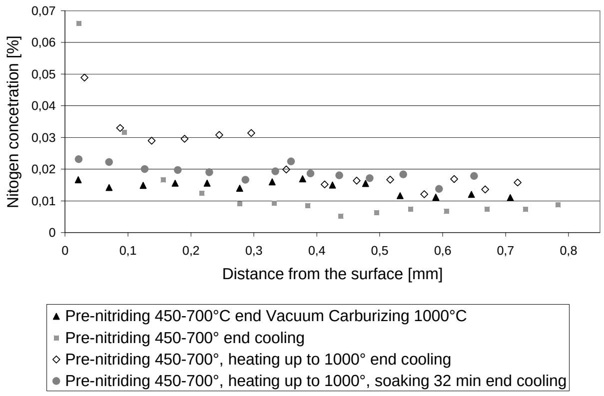


Figure 7 Nitrogen profiles development after various thermal treatments

So little and instantaneous presence of nitrogen before the first boost stage limits austenite grain growth and makes that low pressure carburizing temperature may be increased even to 1000°C without any negative impacts on the microstructure and mechanical properties (figure 8) [13,14,15].

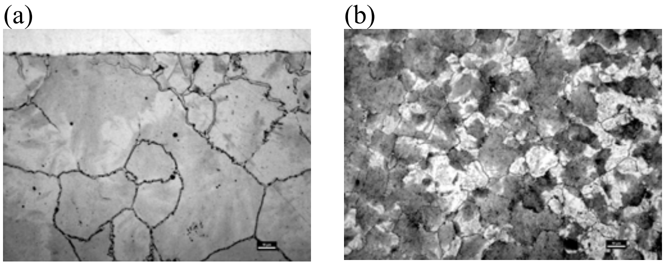

(a) (b)

Figure 8 Effect of the austenite grains growth limitation at 1000°C for 17CrMnNi. Standard LPC (a) and **PreNitLPC®** (b).

This advantage enables the shortening of a high temperature carbon saturation time more than twice, offering an attractive economic technological alternative to classical low pressure carburizing processes. The comparative data has been collected in the table 1.

Table 1. Comparison of low pressure carburizing and **PreNitLPC®** processes

Proces	1	2	3	4
	low pressure carburizing 920°C	PreNitLPC® 950°C	PreNitLPC® 980°C	PreNitLPC® 1000°C
Effective case depth	0,6 mm	0,6 mm	0,6 mm	0,6 mm
Total boost time	23min	17min	12min 39s	11min 26s
Total diffusion time	1h 51min 34s	1h 23min 59s	58min 22s	42min 34s
Total carbon saturation time	2h 14min 34s	1h 40min 59s	1h 11min 01s	**54 min**

All processes carried out accordingly to the parameters from table 1 showed the same carbon profiles, the same extremely high pitting resistance and the same extremely high bending fatigue strength. These results recommend the **PreNitLPC®** processes for the surface hardening of high performance gears and bearings of the new generation.

References

1) P. Kula, J. Olejnik, P. Heilman: Paten No.: EP1558780 (2007), Patent Application No.: US10/531690 (2005),
2) P. Kula, R. Pietrasik, K. Dybowski: Journal of Materials Processing Technology, 164-165, (2005), 876-881
3) W. Dowling et al.: Heat Treatment of Metals.1, (1997) 1-6,
4) Ch. Heming, H. Xieqing, W. Honggang: Journal of Materials Processing Technology 89-90, (1999), 339-343,
5) H. Cheng, H. Xieqing, X. Jianbin: Journal of Materials Processing Technology 138, (2003) 399-402,
6) T. Reti, Z. Fried, I. Felde: Computational Materials Science 22, (2001) 261-278,
7) S. Serajzadeh: Journal of Materials Processing Technology 146, (2004) 311-317,
8) J. Taler, P. Duda: *Simple and Reciprocal Problems in Heat Transfer,* (WNT, Warszawa 2003)
9) S. Wiśniewski, T. Wiśniewski: *Heat Exchange*, (WNT, Warszawa 2000) (In polish)
10) S. Hagi, M. Tamura, M. Okumiya: Patent No.: JP 2004332075, (2004)
11) P. Jacquet, G. Bernard, M. Lambertin: Advanced Materials & Processes, (2000), (Suppl.), H19-H21.
12) P. Kula, J. Olejnik: Patent Application No.: EP1980641 (2008)
13) P. Kula, J. Olejnik, P. Heilman: Paten No.: EP1558781 (2007), Patent Application No.: US10/531477 (2005),
14) P. Kula, D. Siniarski, R. Pietrasik, Ł. Kaczmarek, M. Korecki, A. Adamek: *Proceedings 15th IFHTSE,* (Convention Center Vienna, 2006)
15) P. Kula, D. Siniarski, M. Krasowski: *Proceedings XVIII Physical Metallurgy and Material Science Conferences* (AMT, Warsaw-Jachranka, 2007)