

## Case Hardening in Modern Vacuum Furnaces

PhD, Eng Maciej Korecki, Eng Michał Bazel, Eng Michał Sut – SECO/WARWICK, Świebodzin, Poland  
PhD, DSc, Eng Emilia Wołowicz-Korecka, PhD Sylwester Pawęta – Lodz University of Technology, Poland

### Abstract

The article describes the achievements and application of a new generation of vacuum furnaces into the case hardening technology. The technology is based on low pressure carburizing (LPC) at elevated temperature. Depends on application and productivity there are single-, double-, triple- and multi-chamber furnaces equipped with oil or high pressure gas quenching (HPGQ). Applied vacuum furnaces are fully automatic, computer controlled and equipped with technological support systems in the form

of simulation software SimVaC for LPC and quenching. Presently, vacuum furnaces are capable of handling a number of heat treatment technologies such as: annealing, brazing, sintering, quenching, tempering, carburizing, nitriding, etc. This makes vacuum furnaces versatile, flexible and multipurpose piece of heat and thermo-chemical treatment equipment which ensures high quality, repeatability and reliability at a minimal cost, while cutting down on process time and consumption of utilities and maintaining neutrality to the surroundings and the natural environment.



Fig. 1. Examples of drive transmission parts



### SECO/WARWICK S.A.

street: Sobieskiego 8, 66-200 Świebodzin, Polska  
phone.: +48 68 3820500 | info@secowarwick.com.pl | www.secowarwick.com  
District Court of City Zielona Góra, VIII Department of National Court Register of Polish Companies  
VAT ID No.PL9270100756 | REGON:970011679 | BDO: 00025175 | Commercial Register (KRS):0000271014 |  
Basic Capital: 2 059 710,80 Zł

## Introduction

Case hardening by carburising is one of the most common methods of thermal, or rather thermo-chemical, treatment. It involves diffusive infiltration of carbon from the surface inwards and subsequent quenching of the layer thus formed. This produces a very hard and surface abrasion-resistant element, while at the same time maintaining a flexible core, capable of carrying large, impact loads. Typical examples of implementation of this technology include drive transmission parts, such as wheels, pinions, gears, etc. (Fig. 1).

The case hardening process consists of two phases: carburising, which is typically conducted in endothermic atmospheres, and quenching - typically in oil. It is carried out in atmosphere furnaces: sealed quench furnaces or continuous ones: pusher furnaces and roller hearth furnaces.

The past ten years have seen dynamic growth and the increasing popularity of low-pressure carburising (LPC), commonly called vacuum carburising, which can also be an integral part of the case hardening process. It is carried out in vacuum furnaces: single- or multi-chamber, with oil or high-pressure gas quenching capability. Vacuum furnaces are complete functional and technological replacement for atmosphere furnaces and are available in various configurations for charge (Fig. 2.), semi continuous (Fig. 3.) and mass (Fig.4.) production.

There are several reasons why low-pressure carburising is superior to and replaces traditional carburising. It is a pure and environment-friendly technology, it does not emit heat or harmful pollutants (including GHG).



**Fig. 2. Vector line, single-chamber, LPC, gas quenching vacuum furnace, up to 25 bar**



**Fig. 3. CaseMaster Evolution line, three-chamber, preheating, LPC, oil quench, flow-through vacuum furnaces (semi-continuous)**



**Fig. 4. A modular, multi-chamber, rotary system with LPC and 25 bar gas quenching for mass scale heat treatment**



### SECO/WARWICK S.A.

street: Sobieskiego 8, 66-200 Świebodzin, Polska

phone.: +48 68 3820500 | info@secowarwick.com.pl | www.secowarwick.com

District Court of City Zielona Góra, VIII Department of National Court Register of Polish Companies

VAT ID No.PL9270100756 | REGON:970011679 | BDO: 00025175 | Commercial Register (KRS):0000271014 | Basic

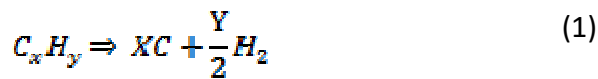
Capital: 2 059 710,80 Zł

It is safe, with no flammable or explosive atmosphere in the furnace chamber and open flame. However, it most importantly guarantees high quality, efficiency and cost-effectiveness. Further considerations will focus on the following areas:

- Quality of carburising
- Uniformity of carburising
- Effectiveness of carburising

### **Low pressure carburizing**

According to the FineCarb® method, low-pressure carburising is carried out in an atmosphere which contains hydrocarbons: acetylene and ethylene, diluted with hydrogen, which is added to slow down the reaction and for better control and purity of the process. The carburising mixture is introduced directly to the work chamber of a vacuum furnace, where it undergoes catalytic decomposition on the surface of parts being carburised; this is accompanied by a depositing of carbon and release of hydrogen, in the following final reaction (1):



Subsequently, carbon from the surface diffuses into the material and forms a layer in accordance with the commonly known law of diffusion. Due to the highly intensive and dynamic nature of the process (saturation of the surface with carbon), the mixture is injected in short periods of a few minutes, with breaks during which carbon diffuses. The boost and diffusion sequence is repeated until the required carbon profile in the layer is formed. The low-pressure carburising process is conducted under the absolute pressure of approx. 5-10 hPa, and the gaseous products of the reaction are removed by a system of vacuum pumps.

Control of the carburising process guarantees its correct outcome and repeatability. Control of an endothermic carburising process is exercised on the basis of the temperature and carbon potential of the atmosphere, usually measured with an oxygen probe, with a dew point meter, or with a CO/CO<sub>2</sub> analyser. Process regulation is effected by adding an enriching gas (methane, propane) in order to increase the carbon potential, or by adding air to reduce it. In low-pressure carburising, controlled parameters include temperature and time sequence, as well as the amount of the mixture of carburising gases. The time and the mixture flow rate are precisely regulated by mass flow controllers, ensuring a high precision of the layer formation and perfect repeatability of the outcome on each part of the charge and across different charges.

### **Intergranular oxidation**

It is an excellent feature of carburising mixture used in an LPC process that it does not contain oxygen in any form, unlike endothermic mixtures which contain a carrier based on reductive gases CO and H<sub>2</sub>, and also oxygen in the form of oxidising compounds: CO<sub>2</sub> and H<sub>2</sub>O, which are present in significant quantities (approx. 1%). They are responsible for the adverse process of intergranular oxidation on boundaries of austenite grains (IGO), which runs down from several to several dozen microns inside the layer, which frequently disqualifies a carburised layer. This phenomenon is absent from the process of low-pressure carburising (Fig. 5).



### **SECO/WARWICK S.A.**

street: Sobieskiego 8, 66-200 Świebodzin, Polska

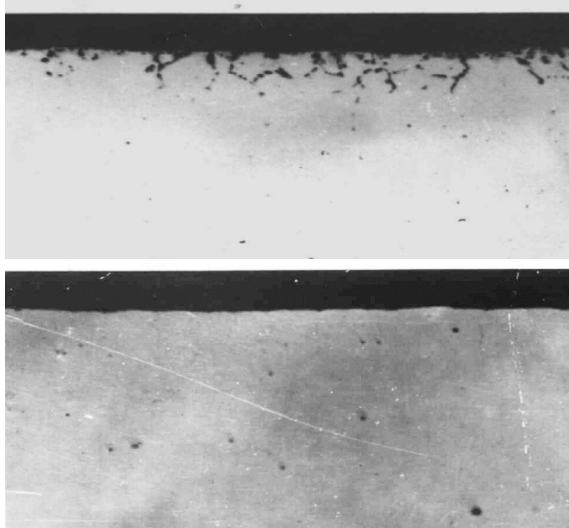
phone.: +48 68 3820500 | info@secowarwick.com.pl | www.secowarwick.com

District Court of City Zielona Góra, VIII Department of National Court Register of Polish Companies

VAT ID No. PL9270100756 | REGON:970011679 | BDO: 00025175 | Commercial Register (KRS):0000271014 | Basic

Capital: 2 059 710,80 Zł





**Fig. 5. The microstructure of a carburised layer: top - after the process conducted in an endothermic atmosphere with visible IGO; bottom**

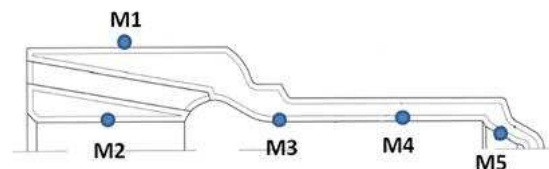
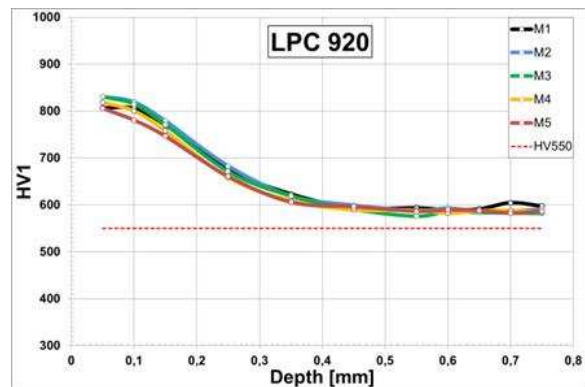
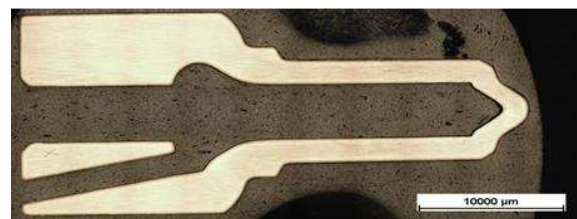
**Carburizing uniformity**

Uniformity of carburising of the whole, densely packed charge, is an important parameter of carburising; this also applies to places to which access is difficult, such as narrow, blind holes. This is a consequence of the penetrating capability and rapidity of exchange of the carburising atmosphere. During the carburising process in an endothermic atmosphere furnace, the atmosphere internal circulation is forced by a convection fan at the rate of several m/s, with limited possibility of penetration of the charge and its individual elements. Whereas, the atmosphere is replaced every 5 minutes (one hot zone volume), with a flow rate of several to several dozen m<sup>3</sup>/h, depending on the size of the working space of the furnace.

The atmosphere in low-pressure carburising is replaced every 5 seconds despite the much smaller demand for the carburising mixture. This is a result of over 100-fold expansion (increase in volume) of gas under pressure of 10 hPa compared to the atmospheric pressure. Also, the mixture spreads practically instantaneously under

low pressure. Gas particles have a speed of several hundred m/s at the outlet of carburising nozzles, which makes them reach each point of the charge space within a fraction of a second.

The mobility of carburising gases during the low-pressure carburising process is several orders of magnitude higher than in the endothermic atmosphere. In consequence, parts with complex surfaces, to which access is difficult, can be carburised perfectly. One of the classic examples is injection nozzles in diesel engines, where uniform carburisation is achieved for thousands pieces, both of the outer surfaces and inside small holes, with a diameter of less than 1 mm and a length to diameter ratio of over 40 (Fig. 6) [1].



**Fig. 6. A uniform carburised layer of the outer and inner layer of a fuel injection nozzle with the hardness profiles after LPC and HPGQ**



**SECO/WARWICK S.A.**

street: Sobieskiego 8, 66-200 Swiebodzin, Polska  
 phone.: +48 68 3820500 | info@secowarwick.com.pl | www.secowarwick.com  
 District Court of City Zielona Góra, VIII Department of National Court Register of Polish Companies  
 VAT ID No.PL9270100756 | REGON:970011679 | BDO: 00025175 | Commercial Register (KRS):0000271014 | Basic  
 Capital: 2 059 710,80 Zł

### Carburizing efficiency

The productivity of the carburising process depends on the availability of carbon from the atmosphere, carbon adsorption on the surface and the rate of its diffusion into the material. Carbon from endothermic atmospheres comes from CO or enriching gases, typically methane or propane. The CO content is approx. 20-30% (and much less C) depending on the method of atmosphere formation; while the content of enriching gases is only a few percent. However, not all of the carbon in such an atmosphere can be used. The size of carbon flux  $J$  from endothermic atmosphere is determined by an equation (2):

$$J = -\beta(C_a - C_s) \quad (2)$$

according to which it is proportional to the carbon transfer coefficient  $\beta$  and the difference between the carbon potential of the atmosphere and carbon surface concentration ( $C_a - C_s$ ). Considering the fact that the reaction driving force is the difference between ( $C_a - C_s$ ), it does not usually exceed 1% (1.2% – 0.2%). It is assumed that the endothermic atmosphere can deliver approx. up to 3 g/m<sup>3</sup> of carbon [2].

Conversely, there is as much as 60-90% of carbon (mass) in the vacuum carburising mixture, which can be used in full. The mixture does not have the carbon potential, but it has the availability of 100%C, which means that a powerful adsorption force is created at the steel surface, driven by the difference between the concentration of carbon available from the atmosphere - 100%C and in the core 0.2%, equalling 99.8%, which is nearly 100 times higher than in the endothermic process. Moreover, a mixture of vacuum carburising gases can supply as much as up

to 600-900 gC/m<sup>3</sup> (depends on a mixture composition), which makes it hundreds times more efficient comparing to endothermic atmosphere and consequently less consumable.

### Carburizing time

Subsequently, carbon adsorbed on the surface diffuses into the steel; this is the phase of diffusion, which does not depend on the carburising atmosphere, but mainly on the properties of the material. In practice, it is the process of carbon diffusion that determines the layer growth rate and, consequently, the effectiveness of the carburising process. Carbon diffusion rate (flux  $J$ ) is described by equation (3):

$$J = D \frac{C_1 - C_2}{dx} \quad (3)$$

which shows that it depends on the difference of carbon concentrations  $C_1 - C_2$  (at the unit distance  $dx$ ) and diffusion coefficients  $D$ . The difference between the carbon concentrations is strongly restricted by the solubility of C in austenite and the limit of carbide formation and it cannot usually exceed 1.5% C. On the other hand, coefficient  $D$  depends strongly on the temperature (function of  $\exp(-k/T)$ ), owing to which the process can be accelerated considerably. Compared to a temperature of 925°C, coefficient  $D$  is higher by about 50% at 950°C, by 100% at 980°C and by 200% at 1020°C. The time needed to form the appropriate carburised layer is reduced at a similar rate, e.g. for 1 mm at 925°C it is 5 h 30 min, at 950°C – 3 h 50 min, at 980°C – 2 h 35 min and at 1020°C – 1 h 35 min (Tab.1). Therefore, an increase in the temperature of carburising (and diffusion) to over 1000°C can greatly reduce the process duration -



### SECO/WARWICK S.A.

street: Sobieskiego 8, 66-200 Świebodzin, Polska

phone.: +48 68 3820500 | info@secowarwick.com.pl | www.secowarwick.com

District Court of City Zielona Góra, VIII Department of National Court Register of Polish Companies

VAT ID No.PL9270100756 | REGON:970011679 | BDO: 00025175 | Commercial Register (KRS):0000271014 | Basic

Capital: 2 059 710,80 Zł

even by as much as 4-5 times compared to the traditional temperatures 920-930°C.

Case depth [mm]	LPC carburising time					
	925°C	950°C	980°C	1000°C	1020°C	1040°C
0.50	1h23m	0h57m	0h39m	0h30m	0h24m	0h19m
1.00	5h30m	3h50m	2h35m	2h00m	1h35m	1h15m
2.00	22h00m	15h10m	10h20m	8h00m	6h10m	4h50m
Time relations	100 %	69 %	47 %	36 %	28 %	22 %

16MnCr5 steel carburising time for the given case depth with criterion of 0.35%C, depending on the process temperature.

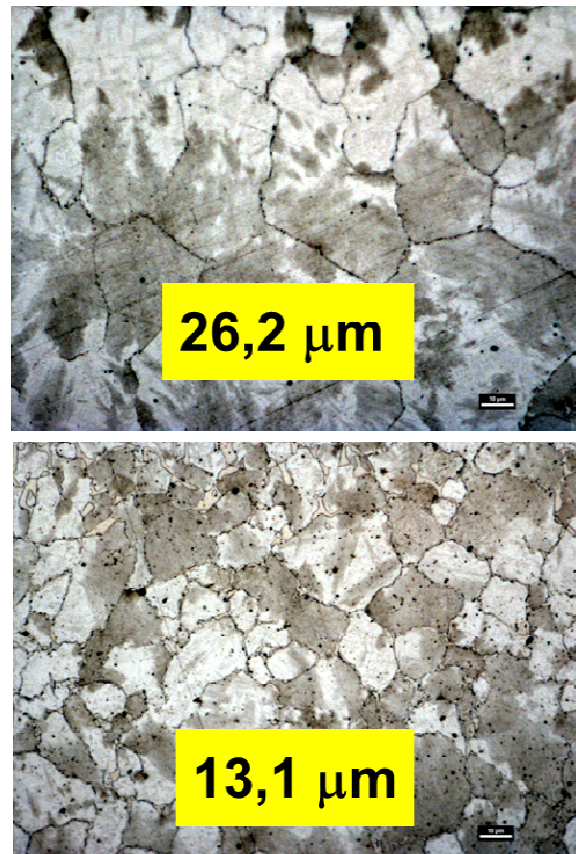
**Tab. 1. Duration of the process of carburizing 16MnCr5 steel depending on the layer thickness and temperature**

The temperature cannot be raised that high in traditional endothermic carburising furnaces due to the dramatic decrease in the life-span of elements of the heating chamber made of heat-resistant alloys, for example a convection fan, heating element, retort, fixture, etc. In such cases, raising the temperature by as little as 50°C can reduce the life-span of critical equipment by half and considerably increase the cost of operation and maintenance.

**PreNitLPC for high temperature carburizing**

This temperature limitations do not exist in vacuum furnaces for LPC, whose standard construction of the graphite chamber makes it possible to reach the temperature of 1200°C and above with no negative consequences. Moreover, the use of equipment made of graphite composite enables one to raise the temperature of carburising freely, the only limitation being excessive growth of austenite grains, which affects some mechanical properties. Excessive growth of austenite grains can be

prevented by PreNitLPC [3]. This method involves introducing nitrogen before the process of austenisation and carburising; by the formation of nitrides, it increases considerably the number of nucleation centres for austenite grains and restricts their propagation by increasing the energy of enlarging grain boundaries. This results in a greater number of smaller grains of austenite, which is shown at the example of carburising 18CrNiMo7-6 steel for a 0.6 mm layer at the temperature of 1000°C (Fig.7.), where the grain size after the PreNitLPC is reduced by half – 13 µm compared to 26 µm grains produced by conventional LPC.



**Fig. 7. The size of an austenite grain following the LPC process for a 0.6 mm layer at a temperature of 1000°C of 18CrMnMo7-6 steel without (top) and with PreNitLPC (bottom)**



**SECO/WARWICK S.A.**

street: Sobieskiego 8, 66-200 Świebodzin, Polska  
 phone.: +48 68 3820500 | info@secowarwick.com.pl | www.secowarwick.com  
 District Court of City Zielona Góra, VIII Department of National Court Register of Polish Companies  
 VAT ID No.PL9270100756 | REGON:970011679 | BDO: 00025175 | Commercial Register (KRS):0000271014 | Basic Capital: 2 059 710,80 Zł



## Case hardening costs

The application of a high temperature during the process of low-pressure carburising and, in consequence, the reduction of its duration, is the basic part of cost reduction for thermal treatment. In general, the effect increases with the thickness of a layer and with the temperature. Tab. 2. [4] presents a comparison of unit costs for the process of endothermic carburising at 930°C in a sealed quench furnace and low-pressure carburising: PreNitLPC carried out at the temperature of 980 and 1020°C, in a CaseMaster Evolution furnace, both of them with the working space with the following dimensions: 900 x 800 x 1200 mm and 650 kg batch net mass (1000 kg gross). Costs refer to total direct and indirect costs including utilities, materials, labour, depreciation, taxes, etc., under conditions of a particular commercial heat treatment shop.

ECD [mm]	ENDO 930 °C [\$/kg]	PreNitLPC [\$/kg]	
		980 °C	1020 °C
0,4	0,89 (100%)	0,92 (103%)	0,99 (112%)
0,6	1,05 (100%)	0,94 (89%)	1,04 (99%)
0,9	1,21 (100%)	1,11 (91%)	1,11 (91%)
1,2	1,52 (100%)	1,31 (86%)	1,22 (80%)
2,0	2,77 (100%)	2,12 (76%)	1,62 (58%)
3,0	4,72 (100%)	3,44 (73%)	2,43 (52%)
5,0	11,77 (100%)	8,11 (69%)	4,86 (41%)

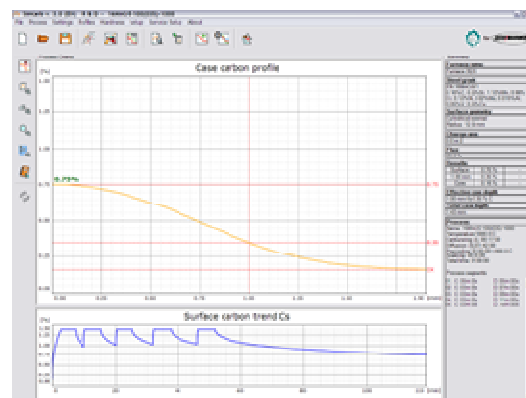
**Tab. 2. Unit costs of case hardening in \$/kg of endothermic and low pressure carburising by PreNitLPC depending on the layer thickness (ECD) and**

The unit costs [\$/kg] are comparable for thin layers, 0,4-0,6 mm, then are brought

down to 80-70% for larger layers at 980°C, and it can be reduced to about 40% from \$12/kg to about \$5/kg for a 5 mm layer at 1020°C.

## SimVaC simulator

The ability to select the right parameters of the carburising process in order to achieve the required carbon (or hardness) profile is of key importance to the quality and productivity of the thermal treatment, especially when the requirements vary from one charge to another. The process outcome can be predicted by means of simulators, both for carburising in atmosphere furnaces and for low-pressure carburising. SimVaC simulator (Fig.8.) can predict precisely the outcome of the low-pressure carburising process in the form of carbon profile (or hardness), and it can use the required profile to suggest the parameters that should be used in the process.



**Fig. 8. The carbon profile simulated by SimVaC for 1,0 mm case depth on the basis of a specific LPC process: 1000°C, only 2 h (17 min boost)**

The simulator takes into account all the elements which affect the process, such as the steel grade, shape, surface area to be carburised, temperature and segmentation of the boost and diffusion phases. A simulation provides a carbon profile and the



## SECO/WARWICK S.A.

street: Sobieskiego 8, 66-200 Swiebodzin, Polska  
 phone.: +48 68 3820500 | info@secowarwick.com.pl | www.secowarwick.com  
 District Court of City Zielona Góra, VIII Department of National Court Register of Polish Companies  
 VAT ID No.PL9270100756 | REGON:970011679 | BDO: 00025175 | Commercial Register (KRS):0000271014 | Basic Capital: 2 059 710,80 Zł

carburising mixture flow rate. The simulator has been verified in thousands of different processes and it guarantees a correct outcome without the need for test runs.

### Summary

Low-pressure carburising is a complete replacement for traditional carburising in an endothermic atmosphere and it is superior to the latter in terms of results, efficiency, productivity and cost-effectiveness. The following advantages are particularly noteworthy:

- No oxidation on the grain boundaries.
- Perfect uniformity of carburising for parts whose surface is difficult to access and densely packed charges.
- High productivity and efficiency of the carburising mixture results in its low consumption.
- Great reduction of the process duration and costs due to the process being carried out at high temperatures.
- High precision of process parameters control as well as precision and repeatability of results.
- Possibility of guaranteed prediction of the process outcome with a SimVaC simulator.

It must also be stressed that both the LPC technology and vacuum furnaces in which the processes are conducted are safe and environmentally friendly. Considering the current trends in industry development, which low-pressure carburising is perfectly in line with, the technology is likely to expand and develop, until its complete domination is achieved.

### References

- [1] M. Korecki, P. Kula, E. Wołowiec, M. Bazel, M. Sut: Low pressure carburizing and nitriding of fuel injection nozzles. Heat Processing 3 (2014) 59-62.

- [2] F. Neumann, U. Wyss: Aufkohlungswirkung von Gasgemischen in System  $H_2/CH_4/H_2O-CO/CO_2-N_2$ . Harterei tech. Mitt 1972, No. 4, p. 253.
- [3] P. Kula, R. Pietrasik, K. Dybowski, M. Korecki, J. Olejnik: PrenitLPC® – The modern technology for automotive. New Challenges in Heat Treatment and Surface Engineering, Dubrovnik, Croatia, 2009
- [4] S. Paweta: Business models of implementation of the efficient low pressure carburizing technology by PreNitLPC. Ph.D. thesis, Lodz University of Technology, 2014



### SECO/WARWICK S.A.

street: Sobieskiego 8, 66-200 Świebodzin, Polska

phone.: +48 68 3820500 | info@secowarwick.com.pl | www.secowarwick.com

District Court of City Zielona Góra, VIII Department of National Court Register of Polish Companies

VAT ID No.PL9270100756 | REGON:970011679 | BDO: 00025175 | Commercial Register (KRS):0000271014 | Basic

Capital: 2 059 710,80 Zł