PROPERTIES OF CASES TREATED BY USING NEW HIGH-TEMPERATURE VACUUM CARBURISATION TECHNOLOGY WITH THE PRE-NITRIDING – PRENiTLPC OPTION*

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Abstract

Vacuum carburising with pre-nitriding (PreNitLPC® technology) involves feeding ammonia during the charge heating stage before carburising. The purpose is to restrict the growth of grain in the surface layer of the carburised steel. The article presents the influence of nitrogen on restricting austenite grain growth during vacuum carburising and on mechanical properties (fatigue strength, impact resistance) of layers that underwent such treatment in comparison with traditional carburising methods.

Keywords: Thermo-chemical treatment; Vacuum carburising; Pre-nitriding; Carburising; Low pressure carburising.

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** Technical contribution to the 70º Congresso Anual da ABM – Internacional and to the 15º ENEMET - Encontro Nacional de Estudantes de Engenharia Metalúrgica, de Materiais e de Minas, part of the ABM Week, August 17th-21st, 2015, Rio de Janeiro, RJ, Brazil.*

1 INTRODUCTION

Carburising continues to be one of the basic thermochemical treatments used for industrial steel. With the development of the industry, carburising has evolved from its simplest forms, such as pack treatment, to advanced vacuum technologies.
At present, vacuum carburisation technology is replacing the older technologies and is more and more widely used, especially in the automotive industry, and most recently it has been applied in the aerospace industry. This stems from a number of advantages that the technology offers; these include the possibility to shorten the carburising time by raising the process temperature, the possibility to use significantly higher carbon potentials for the atmosphere, the elimination of internal oxidisation, clean charge surface after the process, and the reduction of environmentally noxious emissions [1-4].
An important step in the development of this technology is the FineCarb® expert system used for designing vacuum carburised layers which enables selecting optimum process parameters depending on the required hardness distribution in the surface of the treated parts. The system makes it possible to arrive at the optimum composition of the multi-component carburising mix, to control the intensity of gas flow depending on process stage and the surface of the treated charge, and to have continuous control of process parameters on the basis of signals from the exhaust gas monitoring system. FineCarb®has been developed at the Institute of Materials Engineering at the Technical University of Łódź in cooperation with Seco/Warwick S.A. and supports vacuum carburisation furnaces produced by this company [5,6].
The next stage in developing the technological capabilities of the FineCarb® system was creating a carburisation technology supported with pre-nitriding called PreNitLPC®. Research conducted at the Technical University of Łódź made it possible to develop the basics of the technology and prepare it for industrial implementation. This treatment involves introducing ammonia at the initial stage of the process – during the stage of heating for carburising. Owing to this, the layers obtained, which are carburised in higher process temperatures than usual, do not exhibit grain growth. Owing to this, process temperature can be raised even to 1050°C [7,8].
The introduction of nitrogen during the charge heating stage leads to release of nitrides and/or carbonitrides, which constitute the nuclei for austenite grains and block their growth during the carburising stage. This leads to size reduction of the grains in comparison with the gas carburisation technology as well as with vacuum carburisation, thus it makes it possible to intensify the process by raising the treatment temperature without causing the loss of strength properties.

2 MATERIAL AND METHODS

Specimens of 16MnCr5 steel were carburised using three methods: endothermic gas carburisation, vacuum carburisation and vacuum carburisation with pre-nitriding during charge heating stage.

Table 1. Carburizing process conditions.

Type of carburizing	ENDO	LPC	PreNit LPC®		
Process temperature	920°C	920°C	950°C	980°C	1000°C
Thickness layer (criterion 0.4%C)	0.6mm				
Surface concentration	0.75 %C				

The process atmosphere for endothermic gas carburisation was obtained from natural gas while the atmosphere for vacuum carburisation was a mixture of hydrocarbons: acetylene and ethylene (in 1:1 ratio) diluted with hydrogen. In vacuum carburising with pre-nitriding, the nitrogen was obtained from the breakdown of ammonia, which was fed during the charge heating stage in the temperature range between 400 °C and 700 °C at 26hPa. The pressure during vacuum carburising was 3÷8 hPa (pressure fluctuation). The carburising was conducted at various temperatures. The thickness of the layer in each case was 0.6 mm (at a layer criterion of 0.4 %C) and surface carbon concentration 0.75 %C. The processes were designed using the FineCarb® expert system. Detailed process parameters are presented in Table 1.

3 RESULTS AND DISCUSSION

3.1 Shortening Carburising Time

In economic terms, the most effective method for conducting of carburising is to restrict process time to a minimum, especially when the purpose is to obtain thicker layers. This objective can be achieved when process temperature is significantly raised which, of course, may result in the growth of austenite grains. That danger can be prevented through a modification of the vacuum carburisation method by feeding ammonia during the charge heating stage which will cause the release of nitrogen-based, fine-dispersion intermetallic phases that effectively prevent the growth of austenite grains during carburisation.

Table 2. Processes time obtained for different carburizing conditions.

Type of carburizing	ENDO	LPC	PreNi LPC®		
Temperature	920ºC	920°C	950°C	980°C	1000°C
Thickness layer (criterion 0.4%C)	0.6 mm				
Boost time	167min	23min	17min	13min	11min
Diffusion time	-	1h 52min	1h 24min	58min	43min
Total process time	2h 47min	2h 15min	1h 41min	1h 11min	**54 min**

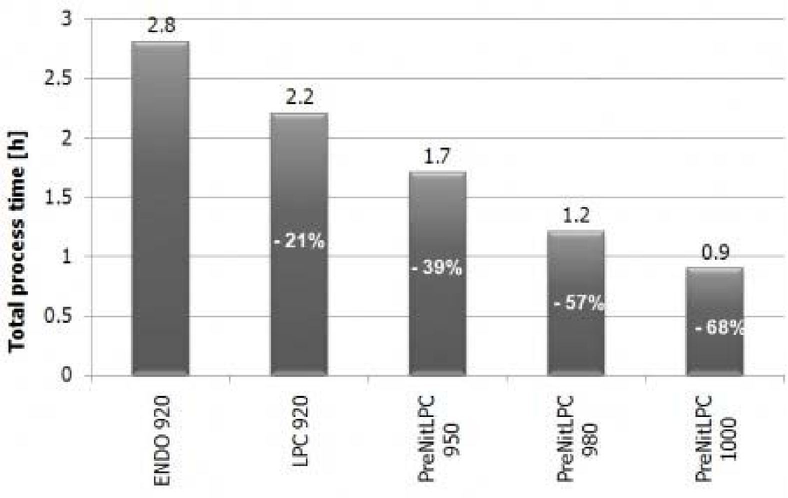

Figure 1. The comparison of total processes time for different types of treatment

When comparing carburising in terms of time necessary to obtain layers of the same thickness (Table 2), it can be established that, as expected, total carburisation time is the shortest at the highest temperature. Namely, at 1000 °C a 0.6 mm thick layer (with the 0.4 %C criterion) can be obtained after just 54 minutes. This represents a shortening of the process by as much as 68% in comparison to endothermic carburisation conducted at 920°C and by 60% in comparison with vacuum carburisation at the same temperature (Fig. 1).

3.2 Estimating Grain Size

In order to estimate the influence of process temperature on the size of austenite grains and the influence of ammonia, the size of former austenite grains was measured (in accordance with the ISO 643:2003 standard). The average grain diameter was measured both in the surface layer as well as in the core of the treated steel. The results are presented in Figures 2 and 3. When comparing individual grain diameters in the core between the various processes, it can be concluded that, just as in theoretical assumptions, the higher the process temperature, the larger the grains in the core. When making the same comparison for carburised layers, it can be concluded that the size of austenite grains created during vacuum carburisation as well as endothermic carburisation at 920°C is the same as in vacuum carburisation at 1000°C with pre-nitriding.

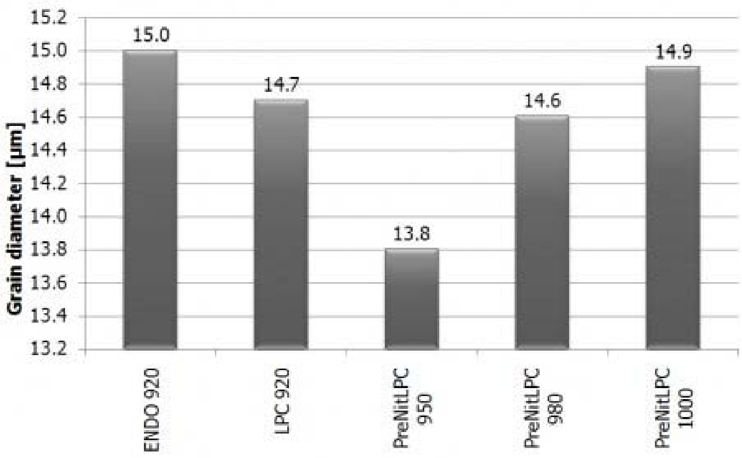

Figure 2. The comparison of the surface layer grain size for 16MnCr5 steel for different types of treatment.

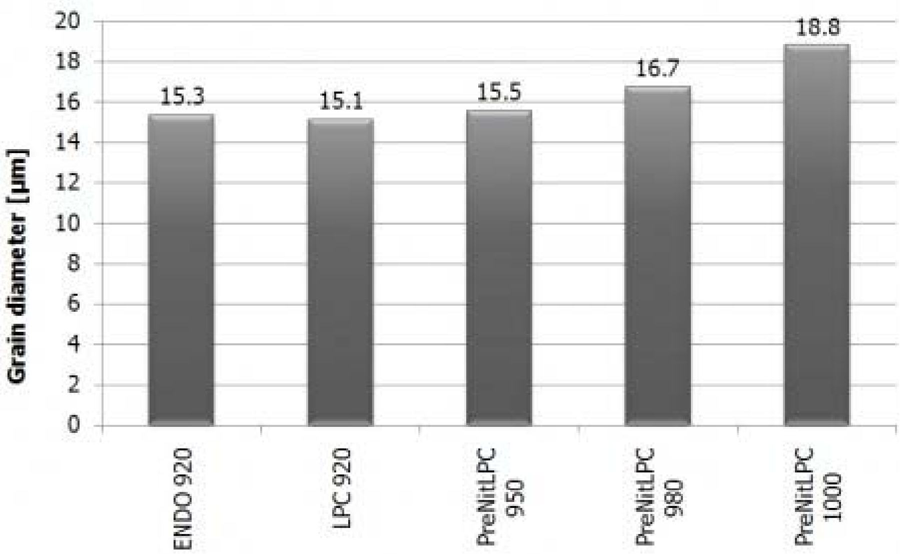

Figure 3. The comparison of the core grain size for 16MnCr5 steel for different types of treatment.

The research has proven that nitrogen does restrict austenite grain size. Therefore, there is a possibility to increase process temperature and thus increase the speed of carbon diffusion, under the condition that mechanical and functional properties, especially fatigue strength, are not decreased.

3.3 Testing Fatigue Strength

In order to evaluate the strength properties of elements treated with the above carburisation methods, a test of fatigue strength was conducted using the high-frequency method. This test was conducted using resonance and the failure point of the tested specimen was defined as vibration frequency change. This method involves measuring the current self-frequency of the tested specimen and using that frequency with an appropriate sinusoidal amplitude. The forced amplitude is adjusted so as to obtain the desired stress at the narrow point of the specimen. The stress value is determined on the basis of the deflection curve. The specimen for fatigue tests was prepared according to the ASTM E 606-04 standard and FEM simulation in order to obtain the optimum stress distribution at the narrow point. The test was conducted at a test station equipped with a Tira vibration exciter TV50101 and

VibrationVIEW controlling amplifier. On the basis of these tests, Wöhler curves were determined in terms of the fatigue limit and endurance limit. The results are presented in Fig. 4. Comparing the fatigue strength of 16MnCr5 steel carburised with different methods, it can be concluded that when vacuum carburisation with pre-nitriding treatment is applied, it does not result in a loss of fatigue strength, on the contrary – it increases the fatigue strength of the tested steel.

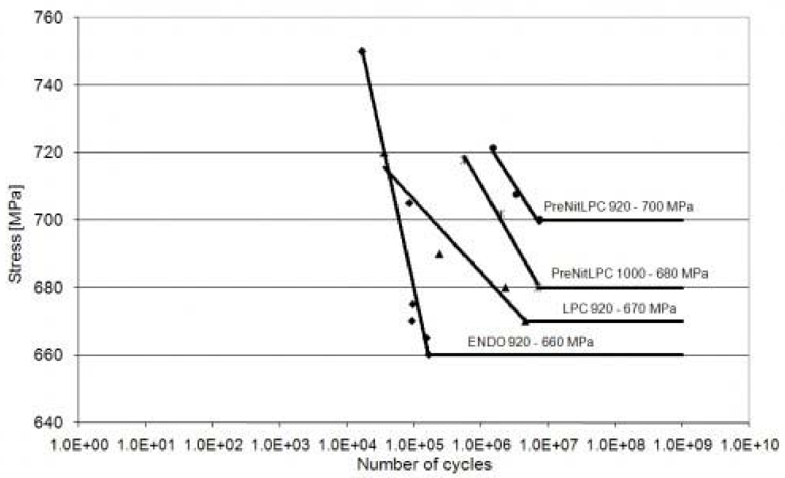

Figure 4. Wöhler's curves within limited and unlimited range of fatigue strength for different types of treatment.

3.4 Testing Contact Strength

The test for contact strength was conducted using a modified four-ball tester (in accordance to the IP 300/82 standard), with a system of three balls and a cone, on layers of 16MnCr5 steel subjected to various types of carburisation, conducted in accordance with parameters provided in Table 1. The results are presented in Fig. 5. The results thus obtained indicate the strength of about 1.6x10-6 cycles with a load of 3924 N, regardless of the carburisation variant used for the specimen.

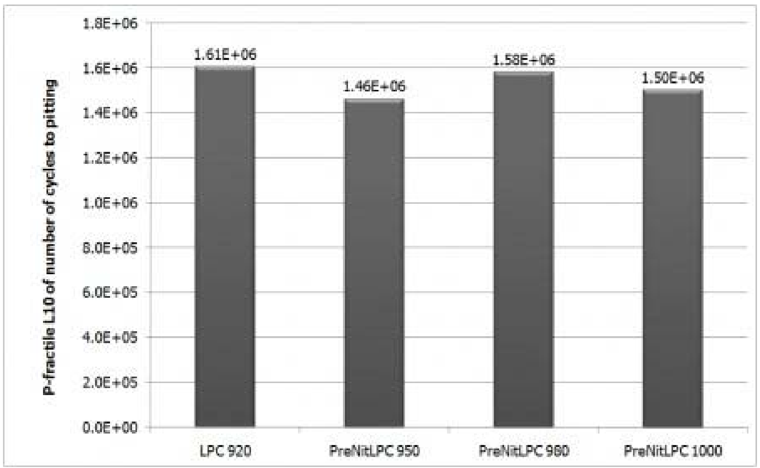

Figure 5. Pitting fatigue strength for 16MnCr5 steel after the low pressure carburising in 920ºC and after the low pressure carburising with the pre-nitriding option in 950, 980 and 1000ºC.

3.5 Impact Test

Measurements of impact strength of 16MnCr5 steel specimens, carburised in accordance with the parameters provided in Table 1, were taken using a Charpy

impact tester (in accordance with the PN-EN ISO 14556 standard). The test was conducted on a u-notched sample; the initial energy of the pendulum was 150 ± 10J. The results are presented in Fig. 6.
The impact strength test of carburised 16MnCr5 steel yields similar values at about 160 J/cm2, regardless of the applied carburisation treatment.

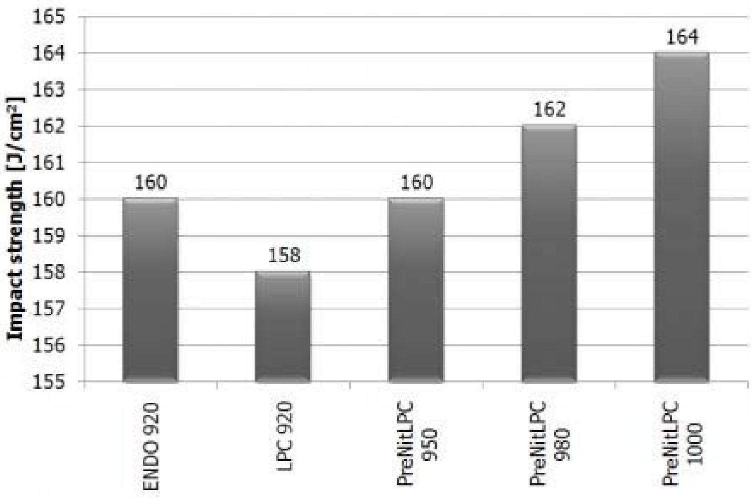

Figure 6. Impact strength (Charpy's test) for 16MnCr5 steel for different types of carburising.

3.6 The Economic Aspect

During the study, a cost analysis was done for the profitability of implementing the PreNitLPC® technology. The total monthly costs borne by a business employing this technology are 1 ÷ 6% higher in comparison with the costs borne by a business that uses an ENDO technology. It must be noted however that due to the differences in process temperatures, and therefore in carbon diffusion coefficients, the time necessary to obtain a given layer thickness is much shorter in the case of the vacuum technology. In consequence, it is possible to complete more processes in a furnace during a set time unit. These differences vary from almost 6% for thin layers up to 100% for thick layers.
The unit cost of carburising 1 kg of charge was calculated using the cost data and the process capacity of the furnace. A comparison of the costs shows the economic advantage of the PreNitLPC® technology. The vacuum technology is more cost-effective even for the thinnest layer of 0.4 mm. In the analysis of unit cost, the vacuum technology is cheaper by over 3% for thin layers, and even up to 48.1% for the thickest layers (for the purposes of this analysis the thickest layer considered was 5 mm).

4 CONCLUSION

To sum up, it can be said that the vacuum carburisation process with pre-nitriding treatment can be conducted at much higher temperatures than those traditionally used for carburisation without the loss of functional properties of the treated elements. Thanks to the higher temperature it is possible to significantly shorten the total process time, which directly translates into a favourable economic result while it does not cause a decline in the mechanical properties of components subjected to this treatment. This opens wide application possibilities, especially for high-quantity series, where the economic aspect is most visible.

Acknowledgments

The authors express their appreciation and thanks to all unnamed companies, employees, students and supporters, who helped to conduct this work and elaborate the paper.

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