ECM Technologies, Large Scale Production Equipment for Heat Treating PM Parts, Improvement and Innovation

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Abstract:

Today Low Pressure Carburizing (LPC) followed by High Pressure Gas Quenching (HPGQ) is an established process to produce high performance components for conventional steels in order to combine high surface hardness and fatigue strength. On the other hand, thanks to its ability to combine good quality and cost saving, PM process is more and more popular in the automotive industry (especially in the transmission applications as synchronizer hubs/rings, sliding sleeves…). In order to answer the increasing application performance requirements, chromium-alloyed grades are often used because they provide a cost-efficient way to reach high mechanical properties. However these materials need specific sintering conditions and cannot be surface hardened by conventional gas carburization and oil quenching. Thus, the alternative LPC & HPGQ applied to PM parts is a very promising, cost-efficient and is an environmental solution to produce high performance parts. This paper presents the last trends and results of this solution.

1. Introduction

Low Pressure Carburizing (LPC) followed by High Pressure Gas Quenching (HPGQ) has been more and more commonly used, especially in the gear industry. This is directly linked to the many advantages of this process compared to the conventional one. These advantages were fully described by Hiller [1] and taken into account both LPC and HPGQ steps, ending to the conclusion that better properties can be obtained with this process adding to the fact that it is cost saving.

Among the technical reasons, one can highlight the main key parameters which make this process very interesting for the Powder Metallurgy (PM) field.

- Firstly, LPC allows a very good control of the atmosphere, free of oxidizing agents, a must in order to avoid the contamination/oxidation of the compacted parts. This is especially true for the Cr containing grades, which are very difficult to case harden without forming chromium oxides. Notice that these grades are more and more popular for PM gears, thanks to the combination of cost issues and good mechanical properties of such alloys. However, they are also more difficult to sinter when using standard belt furnaces as they require specific sintering conditions (temperature/atmosphere).
- Secondly, the use of gas quenching allows a reduction of the part distortion [2] combined with a clean surface, a must for parts containing a remaining slight amount of porosity.
- The possibility to perform the debinding/sintering/LPC/HPGQ (One-Step process) in one process step is a very interesting process route for certain applications.

2. Background and development trends

Since the manufacturing of the first heat treatment furnace in 1928, ECM Technologies is now a privileged partner of most big international automotive and aeronautic groups, and very well established in the LPC field. In order to develop advanced heat treatment furnaces for high performance PM gear applications, ECM Technologies made a partnership agreement with Höganäs, the world leading producer of powder

for the PM field. Consequently, a newly developed furnace has been installed at the Höganäs PoP center (see Figure 1 below) and is used for development of industrial LPC / HPGQ treatments for PM parts.



Figure 1: Vacuum sintering and LPC furnace – PoP center - Sweden

In the PM field, one of the main parameter to be well controlled is the final sintered part's density. It is obvious that the remaining porosity level determines the final mechanical properties compared to a 100% dense part. However, one can say that the arrangement of the pores is more important than the porosity level with LPC treatment. Indeed, the behavior of PM parts during LPC can be strongly modified by the porosity level, especially when percolation occurs. This is directly linked to the amount of active surface area available for the gas cracking and further carbon penetration (see Figure 2).

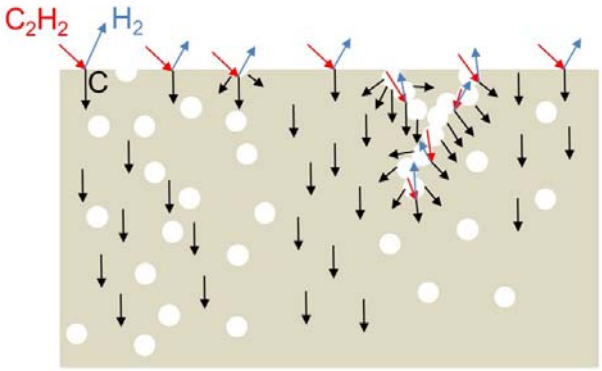


Figure 2: Drawing of surface mechanism

Based on this drawing, it is clear that if the carburizing gas can enters deeply in the material through interconnected pores, carbon enrichment will occur at different depths of the part. As a consequence, the local carbon's contents will be increased compared to the expected level due to the diffusion of the carbon from the surface. The drawback of this phenomenon is the difficulty to keep a good control of the process. Thus, the density level should be selected carefully to avoid the presence of open porosity. If so, the porosity will then affect the process just by increasing a bit the surface area when a pore is located at the surface. This effect is similar to the impact of the roughness which can increase the "active surface".

The effect of the porosity level was studied by Kremel [3] showing that carbon enrichment could be very high for the lowest density level. Over carburizing occurred, resulting in the presence of carbides precipitates or a layer of carbide for the grade containing a carbide forming element (as chromium).
It is established that a density level around 7 is required to minimize the influence of the porosity, and that is why a level of 6.9 to 7.1 has been often used for LPC studies of PM parts [4].

However for a given density level, depending on the porosity shape and/or rearrangement, the amount of open porosity can vary significantly. Therefore, for a density of 7.3, an open porosity level of 4.3% and 0.3% was reported by Santuliana for two different materials [5].

The second parameter of importance regarding LPC treatment is the composition of the steel. It could have an impact on the compaction behavior (and the resulting porosity level as previously discussed), but also on the carbon solubility level in the gamma phase at LPC temperature. Thus, the best results presented in study [4] were reported to be directly linked to the presence of Mo and the consequent increase of carbon solubility. In addition carbide forming element can also have a deep impact on the behavior during LPC treatment. However, this is not a specificity of PM parts and it has already been studied for classical parts. When the carbon limit is exceeded, carbide precipitates, and a carbon deposit can appear [6]. The presence of carbide and/or carbon layer will act as a new carbon source even during the different diffusion steps, ending to a more difficult control of the process.

The third parameter we can play with in PM field is the core carbon content of the grade. This has a huge impact on the final properties of the sintered material as the hardness/toughness compromise is strongly linked to the core carbon content. The effect of carbon content is shown below in Figure 3 in the case of Astaloy 85Mo [7].

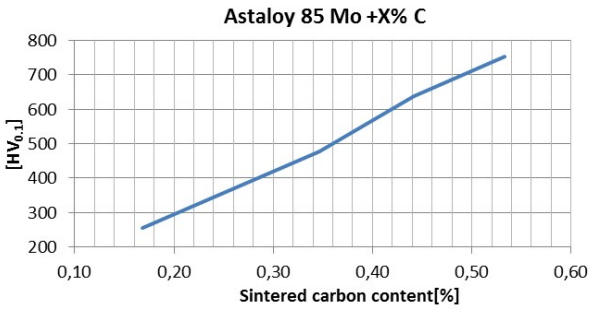


Figure 3: Carbon effect on core hardness

When selecting the right amount of core carbon content, a core hardness around 450 HV0.1 can be reached, which is typically considered as optimum for bending fatigue [8]. Notice that this core carbon content level depends on the grade and could vary for different steel composition.

In order to showcase the benefits of this furnace type on PM parts, we have selected the potential of a process cycle including high temperature sintering directly followed by low pressure carburizing and gas quench (One-Step process). For this investigation we have selected a couple of parameters in accordance to the comments previously presented:

- Concerning the material, a chromium containing grade Astaloy CrM were selected. Such grade is of great interest as high strengths can be achieved at an attractive cost in sinter-hardened conditions. This grade was chosen because of its high Cr content, which sets higher requirements on the sintering conditions and good process control during low pressure carburizing to avoid carbide precipitation.
- A density level of 7.1 g/cc was chosen in order to have some open porosity and it is also a commonly used density level for PM parts.

3. Experimental Results and discussion

The purpose of this study is to demonstrate what can be achieved with an integrated process (One-Step process) with high temperature sintering directly followed by low pressure carburizing at 965˚C. A combination of high surface hardness and high yield strength was sought. Of this reason the core hardness is higher and the case depth is shallower compared to most case carburized materials.

The samples used were tensile and impact energy (ISO 2740 & ISO 5754) test bars. They were compacted to a density of 7.09 g/cc. Sintering and low pressure carburizing was carried out in a two chamber furnace with high pressure gas quench, Fulgura Duo shown in Figure1. 10 mbar pressure was used throughout the entire process with the exception of the quench. During the sintering a partial flow of 1000 l/h of 70%N_2/30%H_2 was used and during the low pressure carburizing a flow of 2000 l/h of 50%C_2H_2/50%N_2 was used. Chemical composition of the mix used in the investigation is summarized in Table 1 below.

Table 1: Chemical composition of the mix.

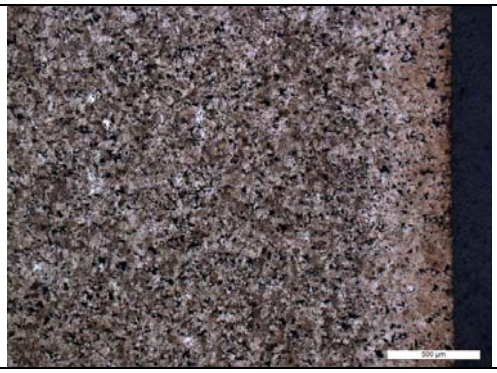
Grade	Mo (%)	Cr (%)	Graphite (%)	MnS [%]
Astaloy CrM	0.5	3.0	0.45	0.30

The pressed samples were divided into 6 series and processed according to the condition in Table 2. Two sintering temperatures were evaluated, with or without a short surface carburization step. Two different quench conditions were evaluated on the samples sintered at 1250˚C.

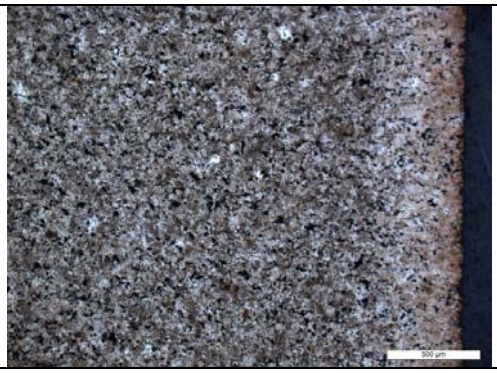
Table 2: Processing conditions

	Sintering [˚C,min]	Quench [bar, fan speed]	Boost & diffusion step	Tempering [˚C, min]
CrM - 1	1180, 30	10, 3600 rpm	No	200, 60
CrM - 2	1180, 30	10, 3600 rpm	Yes	200, 60
CrM - 3	1250, 30	10, 3600 rpm	No	200, 60
CrM - 4	1250, 30	10, 3600 rpm	Yes	200, 60
CrM - 5	1250, 30	3, 2520 rpm	No	200, 60
CrM - 6	1250, 30	3, 2520 rpm	Yes	200, 60

No significant difference in microstructure could be found from the two sintering temperatures except of slightly rounder pore shape after 1250˚C sintering. Therefore, only the metallographic results from series CrM 4 & 6 are presented in Figure 4, 5 and 6.

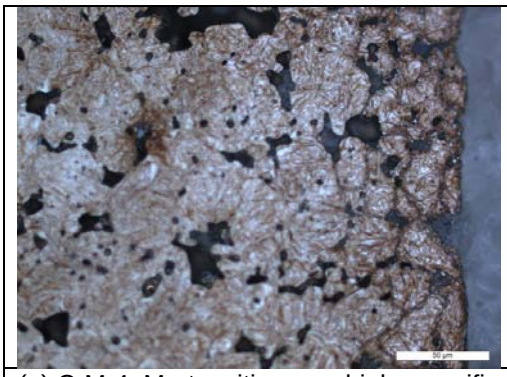


(a) CrM-4: Overview of martensitic case

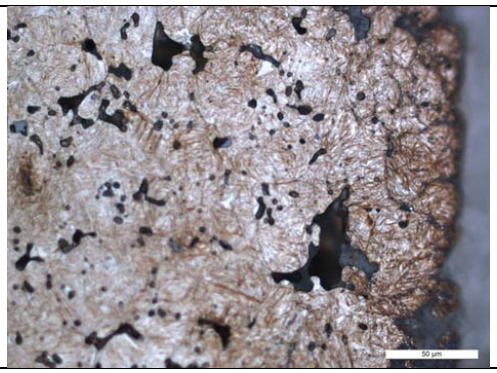


(b) CrM-6: Overview of martensitic case

Figure 4: Overview of hardened case after quench with 10 or 3 bar.

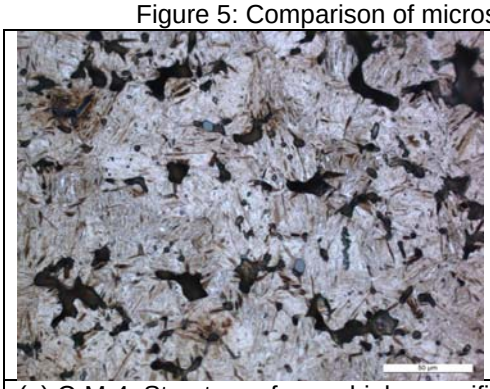


(a) CrM-4: Martensitic case, high magnification

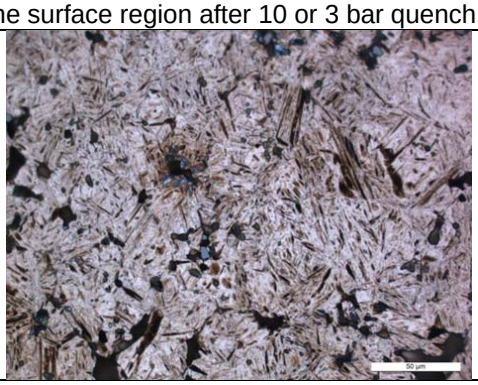


(b) CrM-6: Martensitic case, high magnification

Figure 5: Comparison of microstructure in the surface region after 10 or 3 bar quench.



(a) CrM-4: Structure of core, high magnification



(b) CrM-6: Structure of core, high magnification

Figure 6: Comparison of microstructure in the center after 10 or 3 bar quench.

All the investigated samples exhibited a well-defined harder martensitic outer case, which can be seen in Figure 4. In the very near surface region, minor carbide precipitates were observed in both samples. A small amount of retained austenite was found near the surface of both samples but the difference in cooling rate did not have a significant impact on the amount of retained austenite. In the center, the microstructure was mainly martensitic for both samples, CrM-6 exhibited some low temperature bainite as well due to the lower cooling rate. The micro hardness profiles of the two investigated samples are presented in Figure 7.

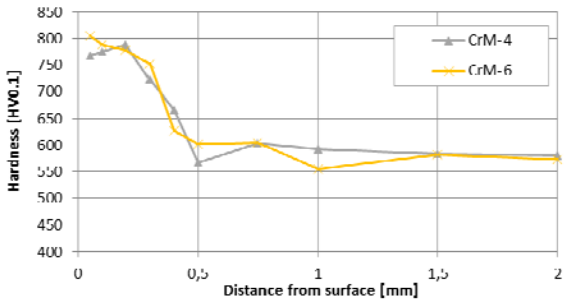


Figure 7: Hardness profile for Astaloy CrM-4 & CrM-6

Overall the microstructure of the investigated samples was as expected, except for the minor carbide precipitation in the near surface region. However, with a slight modification of the low pressure carburizing

parameters this can be avoided, either the boost length or the C_2H_2/N_2 ratio is reduced. The presence of lower bainite in the center of sample CrM-6 should have a slight impact on the micro hardness but this was not observed.

Mechanical properties of the samples are given in Table 3.

Table 3: Measured mechanical properties together with density, carbon and oxygen analysis

	SD [g/cc]	C-content [%]	O-content [%]	IE [J]	HV5	R0.2 [MPa]	Rm [MPa]	A [%]
CrM - 1	7.11	0.37	0.018	22.4	266	996	1111	0.45
CrM - 2	7.11	-	-	10.2	626	-	835	0.11
CrM - 3	7.15	0.35	0.019	24.7	306	1005	1251	0.97
CrM - 4	7.15	-	-	11.3	616	-	879	0.16
CrM - 5	7.15	0.35	0.018	27.0	295	962	1223	0.95
CrM - 6	7.15	-	-	11.3	639	-	915	0.13

By making a slight surface carburization the surface hardness was significantly increased. In most cases more than doubled. As a consequence of the surface carburization, the tensile strength and elongation decreased since the material became harder and more brittle. This same trend was also noticed on the results from the impact testing.
The obtained results are very promising and by changing graphite addition and carburizing process it should be possible to achieve a more ductile material with high enough surface hardness.

4. Conclusions

The right optimization of the material (taken into account the specificity of PM products) combined with the One-Step process allows to produce high performance PM materials.

The interest of such process is substantial for the chromium-alloyed material that requires specific sintering conditions. Thus, the sintering under low pressure avoids any contamination and provides a better sintered microstructure. Thanks to the One-Step process cycle (debinding/sintering/LPC/HPGQ) obtained with the newly developed ICBP® furnace, optimized heat treatment of PM parts can be done in a cost-efficient and environmental way.

High temperature sintering is very beneficial for the mechanical properties of chromium alloyed PM steels. By increasing the temperature from 1180°C to 1250°C tensile strength as well as ductility were improved. Sintering in the ECM furnace worked really well. An indication of good sintering conditions is the very low oxygen content after sintering.

With the alloying system and carbon content selected, the specimens were fully martensitic after the high pressure gas quenching. There was very little difference between the two different settings of pressure and fan speed during the quench. With a larger part this factor would likely be more important.

The core hardness was high as a consequence of the alloying and carbon content. If a more traditional case profile should be obtained a lower graphite addition and somewhat longer boost time would be selected.

References

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