Influence of Alloying Content on Low Pressure Carburized and Gas Quenched PM Components

Magnus Dahlström, magnus.dahlstrom@hoganas.com
Höganäs AB, SE-263 83 Höganäs, Sweden

Abstract

In high strength applications heat treatment is done to obtain the required mechanical properties. Low pressure carburizing + gas quench (LPC + GQ) is one case hardening method that gains interest in the PM industry. Inherent benefits from the LPC + GQ process are less distortions, possibility to case harden chromium alloys and good process control. A trend in the PM industry is to use low alloying content due to benefits in cost and compressibility. However, when comparing alloying content of conventional steel high strength components alloying contents of 2-3% is common. Even if the heat treatment is performed correctly, low hardenability will compromise the final properties of the component.
In this paper the M32 PM transmission gear will be used as demonstrator when influence of alloying content will be presented in regards of processing, microstructure and case profiles. Fatigue strength of investigated materials will be presented and discussed.

1. Introduction

In order to reach the best possible mechanical properties after case hardening it is crucial to optimize the material composition for the intended heat treatment process. Low pressure carburizing and gas quench is a heat treatment process that offers less distortion [1], enables the use of chromium alloyed materials [2], good control of the carburizing process and eliminates the need for secondary washing operations of the treated parts. However, to reach the targeted mechanical properties and microstructure the hardenability of the used material needs to be optimized for gas quench. A trend in the PM industry is to use low alloying content due to benefits in cost and compressibility. However, when comparing alloying content of wrought steel high strength components, alloying contents of 2-3%are common. Low pressure carburizing of higher alloyed materials can present some difficulties if the carburizing parameters are not optimized. Higher alloying content often means that there is lower solubility of carbon in the austenite at carburizing temperature. This requires good control of the surface carbon content throughout the low pressure carburizing process to avoid carbide precipitation. Acetylene is highly active in its pure form but when diluted with nitrogen it is possible to use low pressure carburizing for PM components even at moderate density levels. The same fundamentals that is used to compensate for different density levels can also be applied when low pressure carburizing materials with higher alloying contents to obtain good control of the carburizing process.

In this paper key factors are discussed that influence how to optimize carburizing parameters for a specific density or alloying content and the response it has on microstructure and case profiles. The 4th drive gear for GM M32 gearbox is used as demonstrator when assessing the influence of alloying content on a relevant component. Fatigue strength of chromium alloyed materials heat treated by low pressure carburizing and gas quench is presented.

2. Experimental

Chemical composition of the base powders used in the paper is given in Table 1.

Table 1. Composition of base powders.

Base material	Designation	Mo [%]	Cr [%]	Ni [%]	Mn [%]
Astaloy™ 85 Mo	A-8.5Mo	0.85	---	---	---
Astaloy™ Mo	A-15Mo	1.50	---	---	---
Astaloy™ CrL	A-15Cr2Mo	0.20	1.50	---	---
Astaloy™ CrA	A-18Cr	---	1.80	---	---
Astaloy CrM®	A-30Cr5Mo	0.5	3.00	---	---
Astaloy™ A	A-19Ni5.5Mo2Mn	0.55	---	1.90	0.20
Distaloy™ DC	D-20Ni15Mo	1.50	---	2.00	---

Sintering of all samples was done in a conventional belt furnace in 90% nitrogen and 10% hydrogen atmosphere for 30 minutes at 1120˚C if nothing else is specified.
Low pressure carburizing was done in a two chamber furnace with separate heating / carburizing and high pressure gas quench cell, Fulgura Duo from ECM Technologies. Acetylene diluted with nitrogen to different ratios was used as carburizing gas and the samples were quenched with nitrogen under 20 bars pressure and 3600 rpm fan speed. All samples that were heat treated were positioned in the center of a full load with approximate 160 kg of parts in order to get cooling rates and process conditions feasible also under realistic production conditions. Tempering of the samples was made at 180°C for 60 minutes in air.
The samples used to measure weight gain during low pressure carburizing were pre-treated at 965˚C and 10 mbar pressure with N_2 backfill for 2 hours. This was repeated until the weight of the samples was stable; the samples were then used for measuring the weight gain during a low pressure carburizing cycle. The fatigue strength was evaluated on standardized test bars according to ISO 3928 using the stair case method according to MPIF standard 56.

3. Results and discussion

3.1 Fundamental aspects of low pressure carburizing process of PM steels

The length of the boost steps during low pressure carburizing needs to be optimized for factors such as density, surface condition, alloying content and sintered carbon content. Failure to do so will greatly increase the possibility of over-carburization and carbide precipitation. The amount of carbon that can be dissolved in austenite at austenitizing temperature is highly dependent on the amount and type of alloying elements. This can present a problem during low pressure carburization as there is not the same limit in surface carbon content after each boost step as in conventional gas carburizing (GC), which uses a set carbon potential of the carbon-carrying atmosphere. In low pressure carburizing the process cycle consists of several boost and diffusion steps. During the boost step carbon is transferred into the component surface which then is alloyed to diffuse during the diffusion step. It is the targeted hardness / carbon profile that determines the number of boost and diffusion steps.
An investigation was made with the purpose to study if the rate of carbon pick-up would decrease once the austenite is saturated with carbon. Cylindrical PM samples of A-8.5Mo with 0.20% C and 0.55% C with 7.20 g/cm³ in density were low pressure carburized with one boost step of different length and then immediately quenched. During the boost step a C_2H_2/N_2 flow of 2000/2000 l/h was used. The weight of each sample was measured before and after the treatment. The weight difference corresponds to the amount of absorbed carbon. For reference, a conventional steel sample of AISI 5120 (20MnCr5) was added. The results are shown in Figure 1.

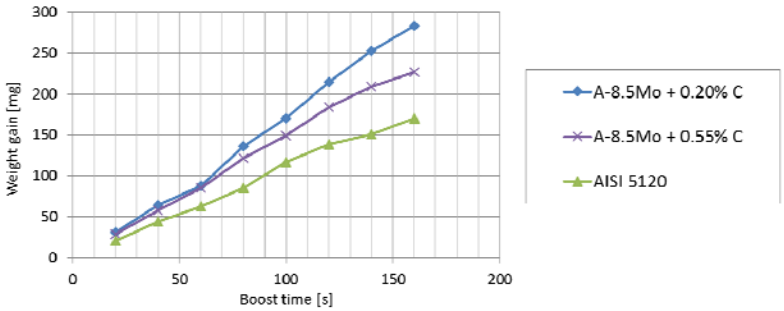
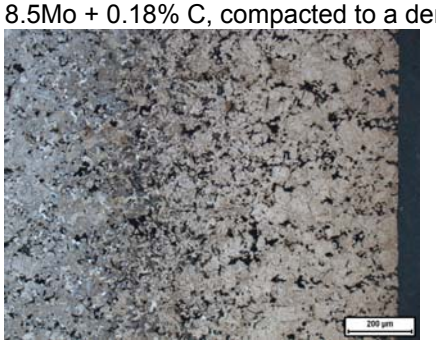


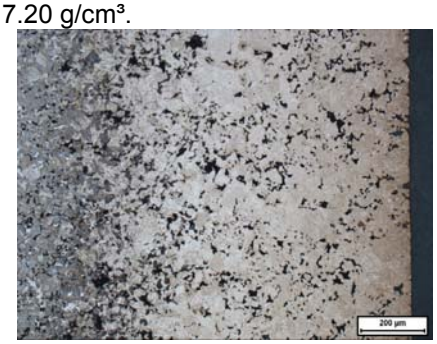
Figure 1. Weight of carbon transferred from atmosphere to component.

A typical length for the first boost of a A-8.5Mo steel with 0.20% C and similar density would be in the range of 50 to 60 seconds using the same carburizing parameters. The results shown in Figure 1 highlight the importance of carefully selected boost lengths in order to avoid carbide precipitation. The component will continue to pick-up carbon at a high rate even long time after carbide precipitation has started. The gradient of the PM sample with 0.55% C follows the 0.20% C initially but decreases after approximate 40-60 s. The slope of the solid steel curve is lower compared to the curves of the two PM samples due to less total surface area available for carbon pick-up.
It is important to remember that the measured weight gain is only valid with the C_2H_2/N_2 gas flow used and in combination with load condition used. Increased C_2H_2 flow will have a strong impact on amount of

carbon that is transferred to the surface. The effect that increased C_2H_2/N_2 ratio will have on the microstructure of the carburized layer is illustrated in Figure 2, were two fatigue bars were low pressure carburized using same boost and diffusions times but different C_2H_2/N_2 ratio. The materiel used was A-8.5Mo + 0.18% C, compacted to a density of 7.20 g/cm³.



a) LPC with 1600/2400 l/h C_2H_2/N_2



b) LPC with 2500/2500 l/h C_2H_2/N_2

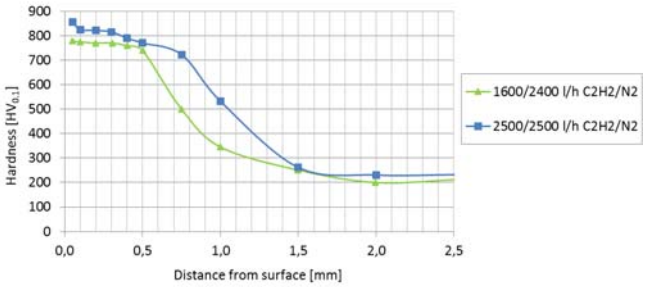


Figure 2. Difference in microstructure and case depth for A-8.5Mo after LPC and GQ with same boost and diffusion time but different C_2H_2/N_2 ratio.

When comparing the measured case profiles of the two samples there is a significant difference in case depth and microstructure. The samples were low pressure carburized with 2500/2500 l/h of C_2H_2/N_2 exhibits somewhat more plate martensite in the surface region, which points to slightly higher carbon content which can be observed in the hardness profile. The results show which impact the used amount of C_2H_2 has on the carbon transfer rate during low pressure carburizing.

In the first study presented in this paper there was a noticeable difference in carbon pick-up for cylinders of solid steel compared to a PM steel at density of 7.20 g/cm³. To further investigate the influence of density in regards of total surface area available for carbon pick-up, the weight gain of cylindrical samples compacted to three different density levels was measured after low pressure carburizing. The low pressure carburizing recipe contained three boost steps with a total carburizing time of 146 s and was optimized for a density level of 7.20 g/cm³. The results are shown in Figure 3.

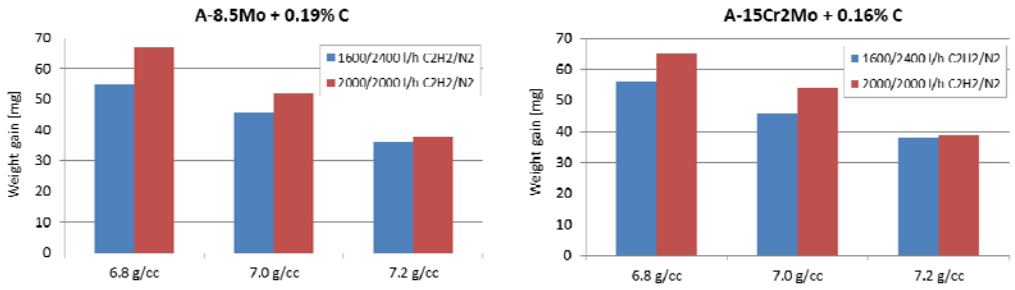


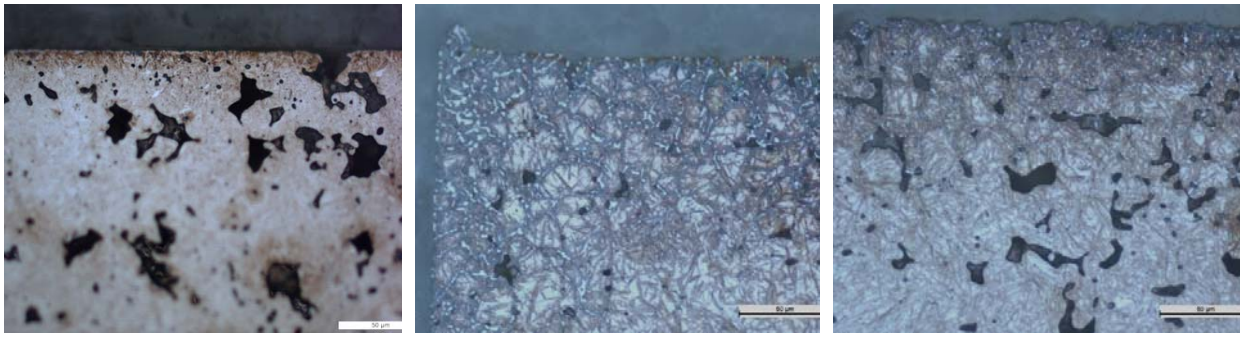
Figure 3. Weight gain of samples after two different used C_2H_2/N_2 ratios.

The results in Figure 3 shows a significant difference in carbon pick-up for the three density levels as can be expected. The effect of the C_2H_2/N_2 ratio on the measured weight gain exhibits a clear density dependency.
With knowledge of how the carbon pick-up is influenced by the C_2H_2/N_2 ratio for different density levels, it is possible to optimize the length of the boost steps for a given component with a specific density. One important factor to consider for materials with high hardenability suitable for gas quenching, is the amount of carbon that can be dissolved in the austenite at carburizing temperature. For such materials, it is important to know the surface carbon content after each boost step in order to avoid carbide precipitation. Examples of solubility of carbon in austenite at 965˚C for a few pre-alloyed material grades is given in Table 2. The values were calculated with Thermo-Calc software [4].

Table 2: Solubility of carbon in austenite at 965˚C for a few selected pre-alloyed materials.

	A-8.5Mo	A-15Mo	A-18Cr	A-30Cr5Mo
Solubility of carbon in austenite γ_{cmax} @ 965˚C [%]	~1,40%	~1,40%	~1.10%	~0.90%

In practice, this means that boost lengths which is optimized for one low-alloyed material might not be applicable for a material with lower solubility of carbon in the austenite at the processing temperature. This is illustrated in Figure 4, were the microstructure of two chromium alloyed materials is presented. The materials are A-18Cr + 0.25% C and A-30Cr5Mo + 0.18% C, which have been low pressure carburized using the same carburizing parameters. The used low pressure carburizing parameters have been optimized for a component of A-18Cr + 0.25% carbon with a density of 7.20 g/cm³.



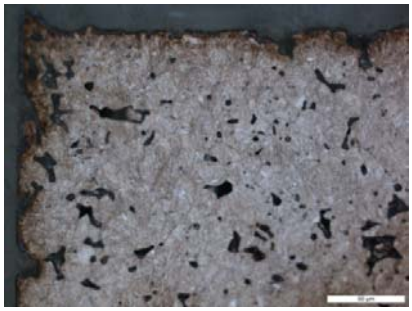
a) Microstructure of A-18Cr, carbide free

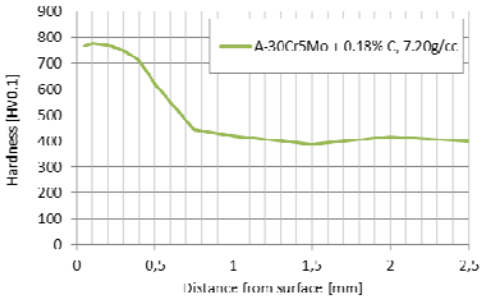
b) Microstructure of A-30Cr5Mo, network of carbides in corner

c) Microstructure of A-30Cr5Mo, carbides in grain boundaries

Figure 4: A-18Cr and A-30Cr5Mo compacted to 7.20g/cc, LPC and GQ using same process parameters.

The component made of A-18Cr exhibits a martensitic case without any carbides while the component made of A-30Cr5Mo exhibits carbide precipitations at the grain boundaries.
There are two options that can be used to low pressure carburize higher alloyed materials to achieve the desired microstructure. Shortening the carburizing time will give a lower surface carbon content after each boost step. However, to ensure good carburizing homogeneity within the load it is advisable not to use too short boost steps. For higher density parts, this will not present an issue but for a more commonly used density level for case hardened PM parts, such as 7.20 g/cm³ there is a risk of affecting the carburizing homogeneity within the charge. As presented earlier the C_2H_2/N_2 ratio has a strong effect on the amount of carbon that is being transferred into the component. By diluting the acetylene with nitrogen, it is possible to lower the activity of the carburizing media and thus achieving same effect as shortening the boost times without jeopardizing carburizing homogeneity in the charge. The same methodology could also be applied on lower density parts to ensure good control of the carburizing process. In Figure 5, a carbide free microstructure of A-30Cr5Mo is shown after low pressure carburizing and gas quenched with a decreased amount of acetylene but maintained total gas flow during the boost step.





a) A-30Cr5Mo + 0.18% C, free of carbides *b) Hardness profile.*

Figure 5. Carbide free microstructure of A-30Cr5Mo + 0.18% C after LPC + GQ.

The results show that it is possible to low pressure carburize also higher alloyed materials, with a relatively low solubility of carbon in the austenite at carburizing temperature, without forming carbide precipitation. This is a prerequisite in order to low pressure carburize and gas quench larger components such as transmission gears.

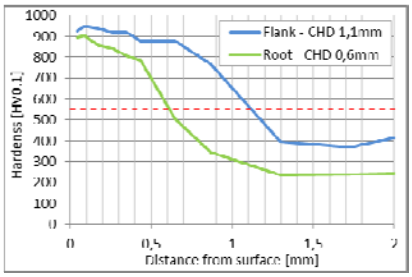
3.2 Low pressure carburizing and gas quench of PM transmission gear.

In order to reach the highest possible mechanical properties after case hardening it is crucial to optimize the material composition for the intended heat treatment. A PM transmission gear was chosen as demonstrator and six different material compositions were low pressure carburized and gas quenched, dimensions of gear is given in Table 3. Main purpose is to identify material compositions that exhibit a core hardness in the range of 400 $HV_{0.1}$, which is considered as optimum for bending fatigue [3]. The results can be seen in Figure 6.

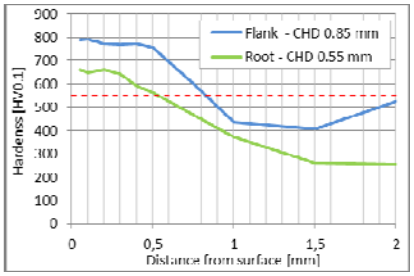
Table 3. Geometrical data and overview of the M32 PM transmission gear.

Outer diameter [mm]	103,6
Inner diameter [mm]	35,3
Gear tooth height [mm]	13,5
Normal module [m_n]	1,64
Weight [g]	460
Density [g/cc]	7,20

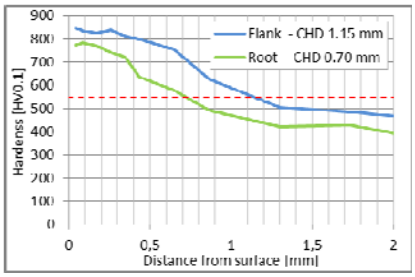




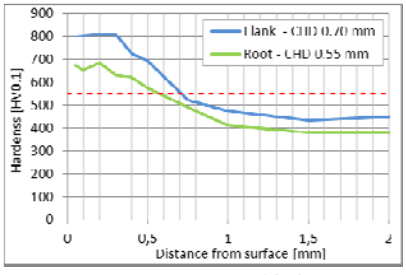
a) A-8.5Mo + 0.26% C



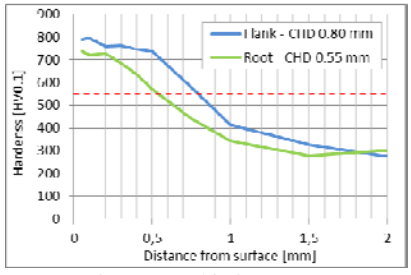
b) A-19Ni5.5Mo2Mn + 0.19% C



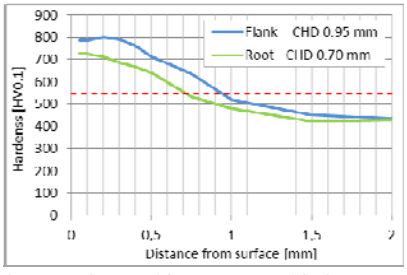
c) A-15Mo + 0.28% C



d) D-20Ni15Mo + 0.19% C



e) A-18Cr + 0.21% C



f) A-18Cr + 2% Ni + 0.25% C

Figure 6. Hardness profiles measured on the M32 PM transmission gears after LPC and GQ.

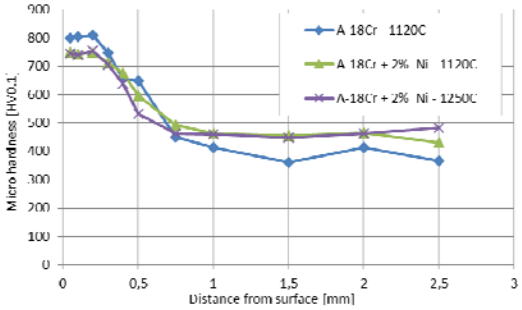
The gear of A-8.5Mo was not tempered before the metallographic investigation, which explains the high surface hardens values. There are some fine-tuning to be done on the carburizing parameters to increase surface hardness in the root for some of the materials. For some of the materials a slight modifications of sintered carbon content is also required to reach the desired core hardness. An indication of that the material is suitable for a component in combination with heat treatment process, is that the difference in hardness between core and tooth-center is not too large. To compensate too low core hardness with increased carbon content will only increase the risk of through hardening the gear tooth. The case profiles from Figure 6a and 6b exhibits a rather significant difference in hardness between tooth center and core. The results from Figure 6 c-f all shows potential to be suitable material compositions for the evaluated gear in combination with low pressure carburizing and gas quench.

3.3 Fatigue

In Table 3, fatigue strength is presented for A-18Cr and A-18Cr + 2% Ni compacted to 7.20 g/cm³. The microstructure of all fatigue tested materials was free from carbides and exhibited a fully martensitic case. Core structure was mainly martensitic with some bainite and nickel-rich austenite in the samples with nickel addition.

Table 3. Fatigue strength of LPC and GQ PM steels together with the hardness profiles.

Material	$\sigma A_{50\%}$ [MPa]	Std dev [MPa]
A-18Cr, 1120˚C	408	< 10
A-18Cr + 2% Ni, 1120˚C	433	< 10
A-18Cr + 2% Ni, 1250˚C	450	< 10



Nickel addition improves the hardenability of the material. The core hardness is increased from approximate 400 to 450 $HV_{0.1}$. Fatigue strength is improved by nickel addition and in combination with high temperature sintering the increase in fatigue strength is significant. The results show that fatigue strength at similar level as for conventional heat treatment can be achieved with low pressure carburization and gas quench. Typical fatigue strength of A-8.5Mo at similar density and case hardened by conventional gas carburizing and oil quench would be in range of 450 MPa($\sigma_{A50\%}$).

4. Conclusions

Low pressure carburizing has been used for a number of different PM materials with successful results. It has been demonstrated that it is possible to low pressure carburize high alloyed materials with rather low solubility of carbon in austenite at carburizing temperature by diluting the acetylene at different degrees. Same methodology can be used to improve the processing control of components of lower densities than 7.20 g/cm³. The ratio between acetylene and nitrogen has a strong influence on the amount of carbon that is transferred into the component.

The results obtained from the low pressure carburized and gas quenched gears shows which hardenability is required for a component of that size.

Fatigue strength data after low pressure carburizing and gas quench have been presented that are at similar levels as after conventional gas carburizing and oil quench.

5. Continued work

The work will be continued to optimize both carburizing parameters and carbon content to achieve good surface hardness in the root as well as core hardness.

An interesting area for future study is to utilize the advantage that gas quench offers in regards of control of cooling rate during quench. In literature, a high cooling rate through the M_s temperature range is often referred to have a negative impact on the residual stresses and fatigue strength [3]. Gas quench enables the possibility to achieve similar or lower cooling rate through this area compared to oil quench.

References

[1] G. Lindell: Vacuum (low-pressure) Carburizing: a user's perspective. Industrial Heating. Sept 2003, Vol. 70, Issue 9.
[2] M. Dahlström, M Larsson, Y. Giraud: High performance PM Components Heat Treated by Low Pressure Carburizing and Gas Quench. Euro PM2013.
[3] Swerea IVF. (2010). Steel and its Heat Treatment - a handbook.
[4] Thermo-Calc Software. www.thermocalc.com