Achievable Properties after Different Heat Treatments of PM Steels

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Abstract:

Heat treatment of powder metallurgy components is often used in order to meet requirements on high hardness and other mechanical properties. Traditional processes such as quench and temper and case carburizing provide a well known route to meet these requirements. Sintered steels behave different compared to wrought steel due to its porous body. Interaction with the carburizing atmosphere for case depth control and decarburization of the surface is important as is the stress relief temperature to achieve required performance. However, sintered PM steels can also be sinter hardened where fast cooling is directly applied after the sintering process in order to form the martensite. In this paper suitable powder metallurgy materials when used at processing routes dedicated for the respective material are investigated with respect to achieved performance after sintering and subsequent heat treatment. Traditional processes such as carburizing and through hardening are selected as well as sinter hardening and low pressure carburizing.

Introduction:

Traditional heat treatment of steel is an old form of improving material performance and has been known for centuries. One good example is the very early knowledge where some tools made of steel, used during the medieval age, could get very hard such as when quenching a red-hot sword by dipping it into water. This is the very early stage of hardening but the mechanisms of getting steel hard by fast quenching were not known by then. However, the steel metallurgy has developed much over time and in particular, the last decade has been very innovative in that respect. Today, the most common processes for heat treatment are the traditional carburizing, carbo-nitriding and through hardening (or neutral hardening). However, very many new processes are used more and more frequently such as low pressure carburizing and various low temperature surface treatment processes such as nitriding, nitro-carburizing etc.

However, heat treatment of PM is more advanced than corresponding processing of solid steels. As PM have a pore system which below densities of approx 7.3 g/cc also are communicating, meaning that exposure to a carbon containing atmosphere goes much faster than is the case for solid steels. Consequently, the heat treatment process is very time dependant and the lower the density is, the shorter the time for processing is. Furthermore, if there is density variations the case depth might also vary within the component. That means that the more complex shape and the lower the density is, the heat treatment must be carefully controlled so too much scatter of component properties is avoided.

In this paper selected powder metallurgy materials, dedicated to be used for different processing routes in heat treatment, are investigated with respect to performance received after sintering and subsequent heat treatment at a density of approx 7.1 g/cc. This can also be a guideline in how to select material and processing route in order to achieve desired material performance based upon available process equipment.

Experimental:

The purpose of the experiments was to recommend suitable materials for defined processing routes so required material/component performance is achieved. The selected process routes were

- sintering and rapid cooling in the same process (sinter hardening)
- sintering and subsequent through hardening (atmosphere treatment + quenching in oil + tempering or vacuum treatment + quenching in N_2 + tempering)
- sintering and subsequent case hardening (carburizing + quenching in oil + tempering or low pressure carburizing + quenching in N_2 + tempering)

Materials for sinter hardening:

For the sinter hardening tests, 2 different materials were selected. These were Astaloy CrM and Distaloy LH. The composition of these powders is according to table 1 below.

Material	Cu (%)	Ni (%)	Mo (%)	Cr (%)	Fe
Astaloy CrM			0.5	3.0	Balance
Distaloy LH	2*	0.9	0.9		Balance
Astaloy CrL			0.2	1.5	Balance
Astaloy 85 Mo			0.85		Balance
Distaloy AE	1.5*	4.0*	0.5*		Balance
Distaloy AB	1.5*	1.75*	0.5*		Balance

* = The alloying elements are here diffusion bonded to the base iron powder

Table 1:   Chemical composition of the materials used in the investigation

Astaloy CrM is selected as this is known to be a good material for sinter hardening [1, 2, 3] and can be used provided the user has good control of the sintering process (endogas can not be used as this will increase the oxygen content too much in the sintered component) and is also a cost effective material.

Distaloy LH is selected as this is a material specially developed for sinter hardening [4] and can be both sintered/rapidly cooled in most atmospheres such as endogas. It is also an economic selection considering the performance achieved.

Materials for through hardening:

For through hardening, 4 various materials were selected. These were Astaloy CrL, Astaloy 85 Mo, Distaloy AB and Distaloy AE. The composition of the powders is according to table 1.

Astaloy CrL is used as high hardness will be achieved to a relatively low cost. Like Astaloy CrM, this material can be used provided the user has good control of the sintering process and is not using endogas. The heat treatment is performed by treatment in a vacuum furnace followed by inert high pressure gas quenching.

Astaloy 85 Mo can be sintered and subsequently heat treated in most atmospheres and gives good through hardened properties.

Distaloy AB has been on the market for a long time and is a suitable Distaloy type of material which can be used for through hardening.

Distaloy AE is a robust material and gives good performance already after sintering but a through hardening operation can further improve performance. Compared to, in particular, the pre-alloyed grades used for heat treatment, Distaloy AE will render in excellent ductility after heat treatment.

Materials for case hardening:

In the case hardening experiments, 3 different grades were selected. These were Astaloy CrL, Astaloy 85 Mo and Distaloy AB. The 2 first materials are pre-alloyed which is recommended to use when a very high, even surface hardness is required. This also gives excellent wear resistance. Other material types often render in various amounts of austenite in the surface after treatment. However, the third material, Distaloy AB, is somewhat higher alloyed but gives a relatively even martensitic structure after heat treatment. The composition of the powders is according to table 1.

Astaloy CrL can be selected provided the user has a good control of the sintering process and is also a good economic selection. A vacuum heat treatment operation will follow the sintering operation.

Astaloy 85 Mo can be sintered and subsequently heat treated in most atmospheres and gives good case hardened properties.

Distaloy AB can be sintered and subsequently heat treated in all types of atmospheres and gives good case hardened properties plus relatively good ductility for being a heat treated material.

Tested materials and procedure for testing:

In table 2 below the mixes used in the different tests are shown.

Mix composition	Designation	Heat treatment
Astaloy CrM + 0.4% C-UF4 + 0.6% Kenolube	CrM-SH	Sinter hardening + tempering
Distaloy LH + 0.85% C-UF4 + 0.6% Kenolube	LH-SH	Sinter hardening + tempering
Astaloy CrL + 0.6% C-UF4 + 0.6% Kenolube	CrL-QT	Sintering + vacuum treatment + quenching N_2 (6 bar) + tempering
Astaloy 85 Mo + 0.5% C-UF4 + 0.6% Kenolube	Mo-QT	Sintering + austenitizing + quenching oil (60ºC) + tempering
Distaloy AE + 0.5% C-UF4 + 0.6% Kenolube	AE-QT	Sintering + austenitizing + quenching oil (60ºC) + tempering
Distaloy AB + 0.5% C-UF4 + 0.6% Kenolube	AB-QT	Sintering + austenitizing + quenching oil (60ºC) + tempering
Astaloy CrL + 0.2% C-UF4 + 0.6% Kenolube	CrL-CQT	Sintering + low pressure carburizing + quenching N_2 (6 bar) + tempering
Astaloy 85 Mo + 0.2% C-UF4 + 0.6% Kenolube	Mo-CQT	Sintering + carburizing + quenching oil (60ºC) + tempering
Distaloy AB + 0.2% C-UF4 + 0.6% Kenolube	AB-CQT	Sintering + carburizing + quenching oil (60ºC) + tempering

Table 2: Materials used for sinter hardening, sintering + through hardening and sintering + case hardening

In order to compare achieved performance for the different processing routes selected, all mixes were compacted to a density of approx 7.1 g/cc and tensile test bars, according to ISO 2740 and plane bending fatigue test bars, according to ISO 3928 (case hardening), were produced. Sintering was performed at 1120ºC for 30 minutes in a 90/10 N_2/H_2 atmosphere with an addition of 0.2% CH_4 in order to assure an even carbon profile and the cooling rate

was in the order of 0.5ºC/s for all of the material combinations that were to be subsequently heat treated in a separate furnace.

All of the sintering tests were performed in one and the same furnace but for the sinter hardening test of Astaloy CrM and Distaloy LH, the cooling rate was adjusted to approx 2 – 3ºC/s. Sinter hardening was followed by tempering at 200ºC for 1 hour in air.

The through hardening tests for Astaloy 85 Mo, Distaloy AB and Distaloy AE were performed by heating to 860ºC for 30 minutes in a separate furnace and then quenched in oil having a temperature of 60ºC. This treatment was followed by tempering for 1 hour at 200ºC in air. For Astaloy CrL, vacuum treatment was performed at 860ºC followed by quenching in high pressure N_2 (6 bar). Tempering was performed at 200ºC for 1 hour in air.

The case hardening experiment for Astaloy 85 Mo and Distaloy AB was performed by heating to 920ºC for 20 minutes at a carbon potential of 0.8%C followed by quenching in oil having a temperature of 60ºC. Tempering was performed for 1 hour at 200ºC in air. For Astaloy CrL, low pressure carburizing was performed at 965ºC and propane was used for carburizing the samples. The Boost carburizing technique was performed and propane was added for 8 minutes followed by a diffusion period of 30 minutes, during next Boost period propane was added for 5 minutes followed by diffusion for 45 minutes. The carbon potential in the atmosphere was approx 1%. Quenching was done in N_2 of 6 bar pressure followed by tempering at 200ºC for 1 hour.

All materials were tensile tested where UTS and A was evaluated. Hardness was measured according to HRC. Fatigue testing was done for the case hardened materials by using plane, four point bending (as-sintered surfaces) and the criteria for fatigue strength was $P_s(50)$ at $N=2x10^6$ stress cycles using the stress ratio R=-1.

Results and discussion:

Sinter hardening:

The tensile strength and elongation of the materials that were sinter hardened can be found in figure 1 below.
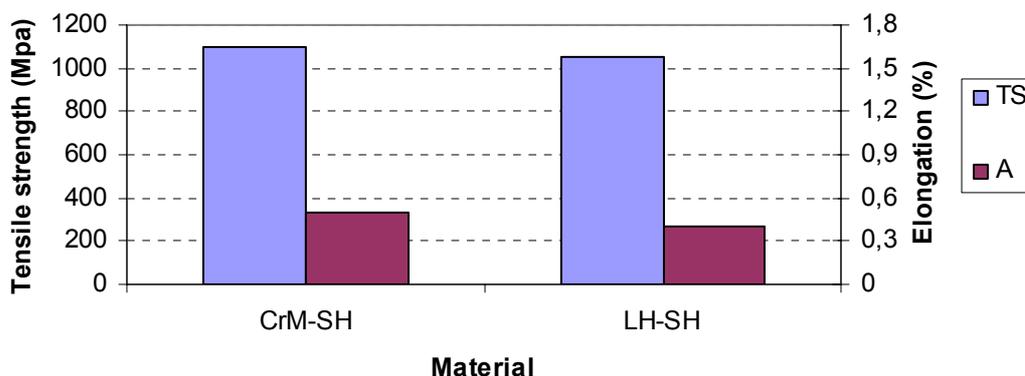

Figure 1: Tensile strength and elongation of CrM-SH and LH-SH after sinter hardening

Both of the materials have a tensile strength exceeding 1,000 MPa. The relative high strength achieved with CrM-SH, when considering the low carbon content, is due to that we have a fine disperse carbide formation in the material before transforming the rest of the structure to martensite. The combination of finely dispersed carbides and martensite gives a

very high strength structure. In the case of LH-SH, the high carbon content makes it a good material for hardenability and corresponding high strength of the martensite after sinter hardening although carbide precipitates does not appear in the material as is the case for CrM-SH.

The ductility of these materials is relatively good when considering that a sinter hardening operation is performed

Sinter hardening results in relatively large grain sizes and large grain size normally gives very low ductility. Therefore, it is of utmost importance that sinter hardened materials are tempered after the sinter hardening process. This is, however, not always done in practical operation.

The hardness of martensite is very much related to the carbon content but in the case of CrM-SH, the hardness is higher than expected even for a relatively low content of carbon in the material. The hardness after sinter hardening of CrM-SH and LH-SH is showed in figure 2 below.
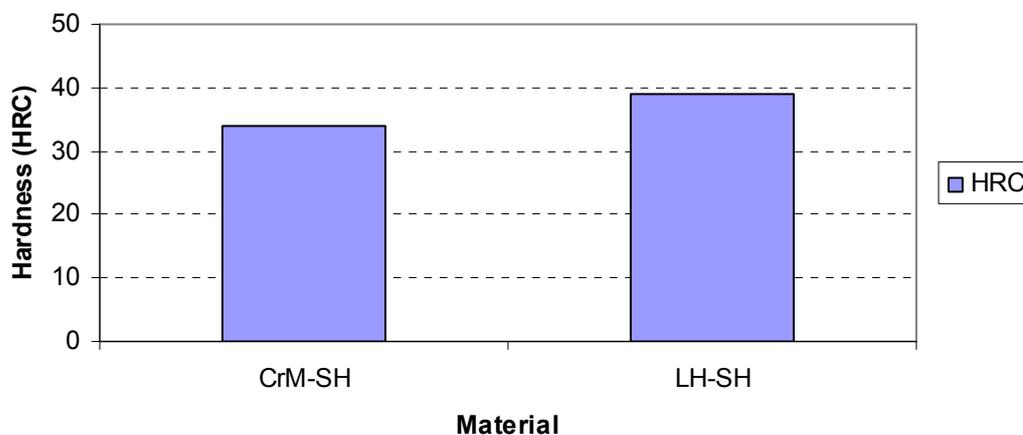

Figure 2: Hardness of CrM-SH and LH-SH after sinter hardening

The higher hardness for LH-SH compared to CrM-SH is related to the higher carbon content in LH-SH compared to CrM-SH. Actually, the hardness contribution from precipitation hardening of very small carbides in CrM-SH can not contribute to such an extent to hardness that it will be in parity of the hardness for LH-SH with the high carbon content.

Discussion sinter hardening:

Sinter hardening is a good alternative to sintering and a subsequent through hardening operation when producing high performance components provided we are dealing with relatively simple geometries and not too heavy components. Complex shapes often renders in density variations within the components which can be tricky to retain without too high costs involved. Too heavy components can get too large variations in cooling rate in the sintering process so various material micro structures appear in different areas of the component. Consequently, distortions might occur and a tricky machining operation is necessary to retain required tolerance of the component.

However, the hardenabilities of Astaloy CrM and Distaloy LH respectively are that good so well controllable cooling rates (that are relatively low) can quite easily transform produced components in a desired direction during the sinter hardening process. Therefore, probably also relatively complex- and heavy components should be possible to produce economically with such material types provided the process control is good.

Through hardening:

The tensile strength and elongation of the materials that were sintered and subsequently through hardened can be found in figure 3 below.
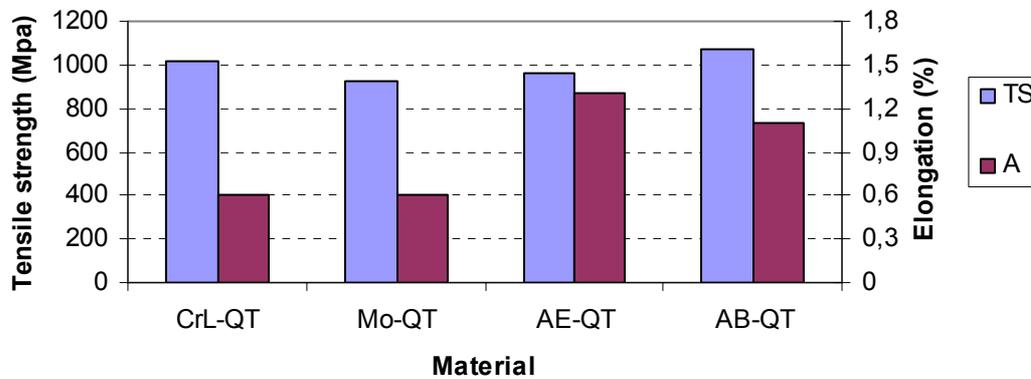

Figure 3: Tensile strength and elongation of CrL-QT, Mo-QT, AE-QT and AB-QT after sintering and subsequent through hardening

All materials exhibit a tensile strength that exceeds 900 MPa after sintering and subsequently through hardening. It can be noted that CrL-QT gives a higher tensile strength than Mo-QT although both of them appear in a sintered state with an upper bainite structure. However, after heat treatment it might be so that all of the fine dispersed Cr carbides in CrL-QT does not dissolve totally in the austenite and a combined effect of martensite and fine precipitates of Cr-carbides will give the extra strength of the Cr alloyed material compared to the Mo alloyed material.

AE-QT gives a relatively high strength but can not match the best material in tensile strength. The reason is that the entire structure does not transform into martensite but there are other advantages to use this material as-heat treated which will be discussed later. AB-QT, on the other hand, gives good strength and this is associated to a high content of martensite in the structure and it is also relatively highly alloyed compared to CrL-QT and Mo-QT. Therefore, very good strength is achieved for AB-QT.

The heat treated Distaloys gives excellent ductility compared to the pre-alloyed materials and the reason is that the entire structure is not martensitic and can allow some deformation before fracture. Therefore, if ductility is necessary for the function of the component, the Distaloys are good selection of material.

Somewhat better ductility is found for the pre-alloyed grades after sintering and subsequent through hardening than for the sinter hardened materials. This is probably due to that the grain size has been refined by re-heating to the austenite area prior to hardening in a separate unit.

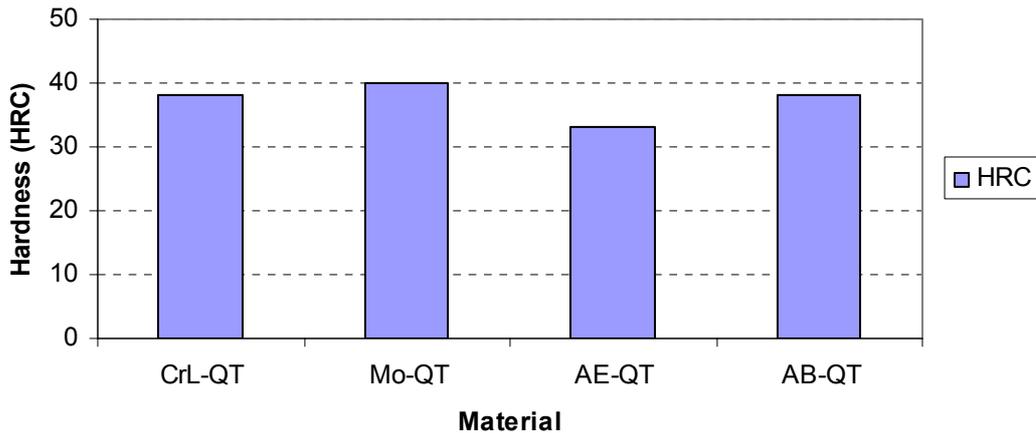The hardness of the materials is found in figure 4 below.

Figure 4: Hardness of CrL-QT, Mo-QT, AE-QT and AB-QT after sintering and subsequent through hardening

Here, it can be notified that all the materials, except AE-QT, achieve HRC figures in the order of 40, the relatively lower figure for AE-QT is associated to the content of austenite and some bainite in the structure that is found after through hardening. Therefore, for e g heavy wear applications, it is advisable to select AB-QT instead of AE-QT.

Discussion through hardening:

Sintering and subsequent through hardening is a good process selection if good wear resistance is important in the application and if it is not considered possible to sinter harden the material because of too complicated shape and/or heavy weight of the component. Here, relatively low alloyed materials can be selected as the high cooling rate during quenching in oil will transform the matrix to martensite. However, if good wear resistance is not critical for the application, Distaloy AE can be an alternative as this material is very robust during sintering and that good ductility/impact properties are achieved after the heat treatment operation.

Case hardening:

The tensile strength and elongation of the materials that were sintered and subsequently case hardened is found in figure 5 below
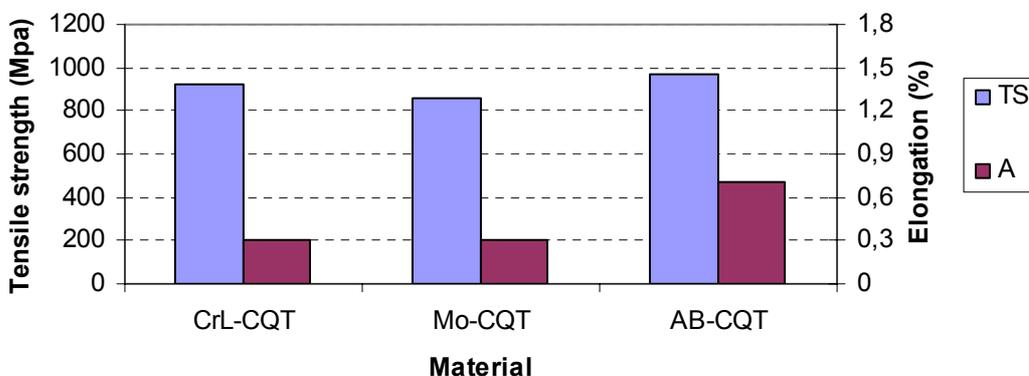

Figure 5: Tensile strength and elongation of CrL-CQT, Mo-CQT and AB-CQT after sintering and subsequent case hardening

Here it can be notified that the strength figures for CrL-CQT and Mo-CQT is somewhat lower than is the case of the corresponding through hardened materials. The reason is the stress distribution of the cross section that is more favourable, as regards tensile properties, for through hardened materials than for case hardened materials. Although there is compressive stresses at the surface, cracks are more easily initiated in the centre of the material where there are tensile stresses present. AB-CQT exhibits high strength and is superior to the pre-alloyed grades.

The ductility is somewhat lower than for the through hardened materials but still there is relatively good ductility in AB-CQT. The reason for it is the presence of some austenite which has a positive effect on ductility.

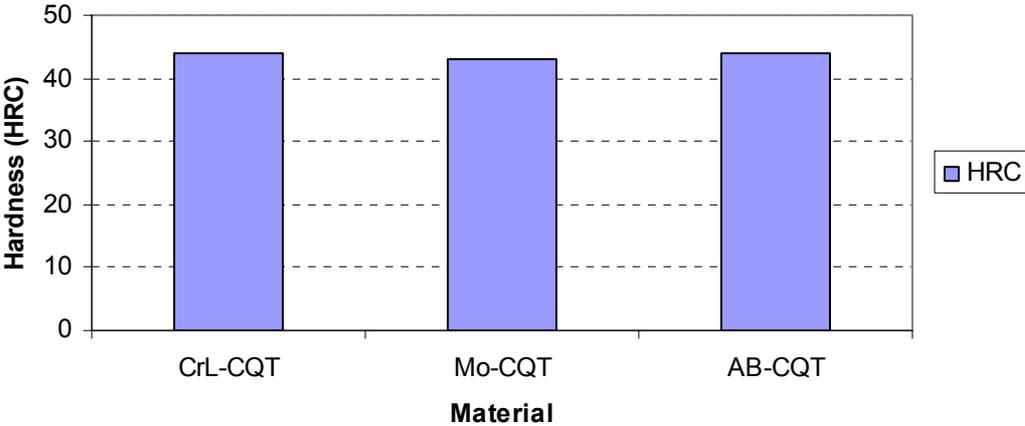The hardness figures are presented in figure 6 below.

Figure 6: Hardness of CrL-CQT, Mo-CQT and AB-CQT after sintering and subsequent case hardening

For all materials, there is quite similar surface hardness and these materials are good for wear resistance but also for fatigue strength due to a favourable stress situation at the surface. Compressive stresses will deteriorate fatigue crack initiation and a less favourable stress situation at the centre of the components has quite low significance, in particular regarding bending performance which is a very common stress state of fatigue loaded PM components.

In figure 7 below the fatigue performance of the case hardened materials is showed.
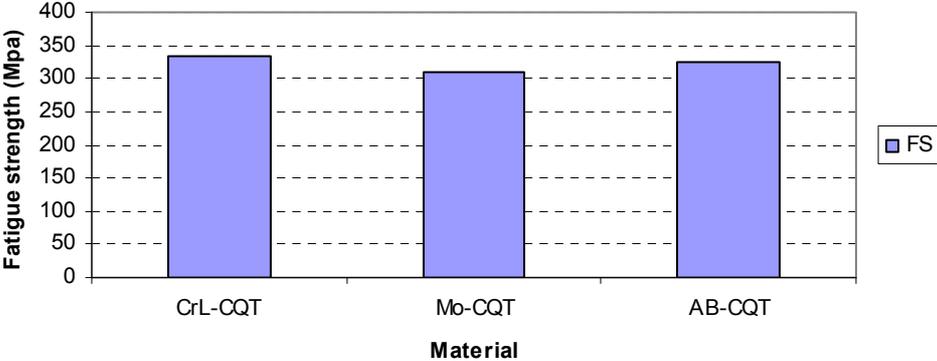

Figure 7: Fatigue performance of CrL-CQT, Mo-CQT and AB-CQT after sintering and subsequent case hardening

All materials will exhibit fatigue properties exceeding 300 MPa. This is, as also has been mentioned above, a result from a favourable stress situation that is found at the surface. CrL-CQT and AB-CQT show somewhat superior fatigue strength as compared to Mo-CQT.

Discussion case hardening:

Sintering and subsequent case hardening should in particular be selected when processing applications requiring high wear resistance and fatigue properties. Pre-alloyed materials are selected for this purpose but AB-CQT can also be selected as the alloying content is not too high which otherwise will result in some austenite at the surface. The Distaloy also provides some ductility.

Concluding remarks:

Heat treatment of PM materials can result in very good properties provided suitable materials and a correct process route is selected.

Sinter hardening demands relatively much of alloying elements in solid solution so reasonable cooling rates can transform the structure into martensite. Therefore for PM materials alloyed with Cr and/or Mo are suitable alloying elements as these have a pronounced effect upon hardenability. CrM-SH (Astaloy CrM) and LH-SH (Distaloy LH) are 2 good examples of sinter hardenable grades.

Sintering and subsequent through hardening can easily manage low alloyed material compositions. CrL-QT (Astaloy CrL) is a good selection provided correct process conditions are used and can be an economic alternative to Mo-QT (Astaloy 85 Mo). However, sufficient hardness is achieved with Mo-QT which also is easy to process. AB-QT (Distaloy AB) gives good properties and AE-QT (Distaloy AE) should be used if high ductility is desired.

Sintering and subsequent case hardening should use low alloyed compositions. Mo-CQT (Astaloy 85 Mo), CrL-CQT (Astaloy CrL) and AB-CQT (Distaloy AB) are all good candidate materials. In particular, for highly loaded fatigue applications, a process route including a case hardening operation should be selected.

References:

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[4] Howe, I et al. "Performance Characteristics of Newly Developed Ni-Mo Sinter-hardened PM Steel", Advances in Powder Metallurgy & Particulate Materials, compiled by Roger Lawcock et al., MPIF, 2008, part 7, pp 250 - 263