COST EFFECTIVE MATERIALS FOR SINTER HARDENING APPLICATIONS

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ABSTRACT

Sinter hardening is a well established industrial process which combines sintering and quenching in one step, and thereby avoiding re-heating and quenching of parts. It is getting an ever increasing interest thanks to availability of cost effective low alloy steel powders, which realize a compromise between compressibility and hardenability, on one side, and to the development of continuous furnaces which allow cooling rates high enough to transform austenite into martensite in these low alloyed steels. In this paper both static and dynamic properties obtained with a number of cost effective low alloy steel powders after sinter hardening will be presented.

INTRODUCTION

The development of new materials and cost effective processes are two main factors that have contributed to the growth of PM during the last decades. Sinter-hardening , which utilises high performance materials in combination with an accelerated post sintering cooling rate, is one example of such material and process development (1,2,3). As this technique is a one step process it combines good manufacturing economy with the ability to achieve a unique combination of mechanical properties.

By controlling the post sintering cooling rate, the microstructure can be manipulated to form the required amount of martensite to obtain the desired mechanical properties.. By increasing the hardenability of the material still large amounts of martensite can be obtained also at lower cooling rates. Alloying elements such as molybdenum, chromium, nickel and copper all promote hardenability. In recent years there has been a dramatic rise in raw material costs of especially molybdenum, nickel and copper. These are typical alloying elements used in applications utilizing traditional sinter and sinter hardened PM materials. Therefore there is a continuously growing interest to utilize more cost effective alloying elements such as chromium and leaner nickel /molybdenum alloyed steels.

In this paper the influence of alloying system and cooling rate on both microstructure and mechanical properties will be discussed. Besides composition and microstructure, density and mass play an important role to achieve the desired properties. Therefore properties after sinter hardening were evaluated at two density levels, 7.0 $glcm^3$ and 7.15 $glcm^3$. The influence of mass on the resulting microstructure and hardness after sinter hardening will also be discussed.

EXPERIMENTAL PROCEDURE

Materials

In order to compare the sinter hardening performance of two pre-alloyed Cr alloyed PM steels and two new cost effective Ni/Mo steels with a conventional Ni/Mo steel, FLC 4608, five different powder mixes were prepared. The base powders used for the mixes were Astaloy A (FL-4600), Astaloy CrL (FL-5208), Astaloy CrM (FL-5305), Astaloy LH and D. LH. The chemical composition of these base powders is shown in Table 1 below. Astaloy A is a fully pre-alloyed powders with 1,9% Ni and 0,5% Mo. Astaloy CrL is a fully pre-alloyed powder containing 1,5% Cr and 0,2% Mo whereas Astaloy CrM is pre-alloyed with 3% Cr and 0,5% Mo. Astaloy LH is a new fully pre-alloyed powder with 0,9% Ni and 0,9% Mo. The D. LH powder is based on the fully pre-alloyed Astaloy LH to which 2% Cu has been diffusion alloyed.

Table 1: Chemical composition of the investigated base powders.

Grade	**MPIF code**	**Ni %**	**Mo %**	**Cr %**	**Mn %**	**Cu %**
Astaloy CrL	FL-5208		0.2	1.5	0.2	
Astaloy CrM	FL-5305		0.5	3.0	0.2	
Astaloy A	FL-4600	1.9	0.5		0.2	
Astaloy LH		0.9	0.9		0.2	
D. LH		0.9	0.9		0.2	2.0*

* Diffusion bonded

The compositions of the mixes prepared are shown in Table 2. The identification codes for these mixes used in this paper are also shown in this table. 0.60 % Kenolube was added as lubricant. The copper admixed to the different was -325 mesh electrolytic copper powder, and the graphite used in all cases was UF-4 from Kropfmühl.

.Table 2. Compositions of the powder mixes used.

Base powder	**Identification code**	**Ni %**	**Mo %**	**Cr %**	**Cu %**	**Gr. %**	**Lub. %**
Astaloy CrL	CrL		0,2	1,5	2,0**	0,85	0,6
Astaloy CrM	CrM		0,5	3,0		0,45	0,6
Astaloy A	4608	1,9	0,5		2,0**	0,85	0,6
Astaloy LH	ALH	0,9	0,9		2,0**	0,85	0,6
D. LH	D. LH	0,9	0,9		2,0*	0,85	0,6

*Diffusion bonded ** Admixed

Processing conditions

The specimens, tensile test bars, impact energy bars and plane bending fatigue specimens according to ISO standards were compacted in a 100 ton, semi-automatic laboratory press, to two different density levels, 7.00 g/cm^3 and 7.15 g/cm^{3}by conventional compaction. It is important to notice that the compaction pressures required to obtain the desired density levels will vary considerably with the base material selected. The compressibility of the different base powders are shown in figure 1.

Sintering was carried out in a laboratory sintering furnace from Cremer equipped with rapid post sintering cooling system to be able to accelerate the cooling capabilities of the continuous belt furnace to the desired cooling rate. The sintering temperature used was set to 1120 ºC. Sintering time was 30 min at temperature and the atmosphere used was 90/10 mix of nitrogen

nitrogen and hydrogen. Two different cooling rates were used, 0,8ºC/s for reference and 2,5 ºC/s for sinterhardening. After sintering tempering was carried out at 200º C for 60 minutes in air.

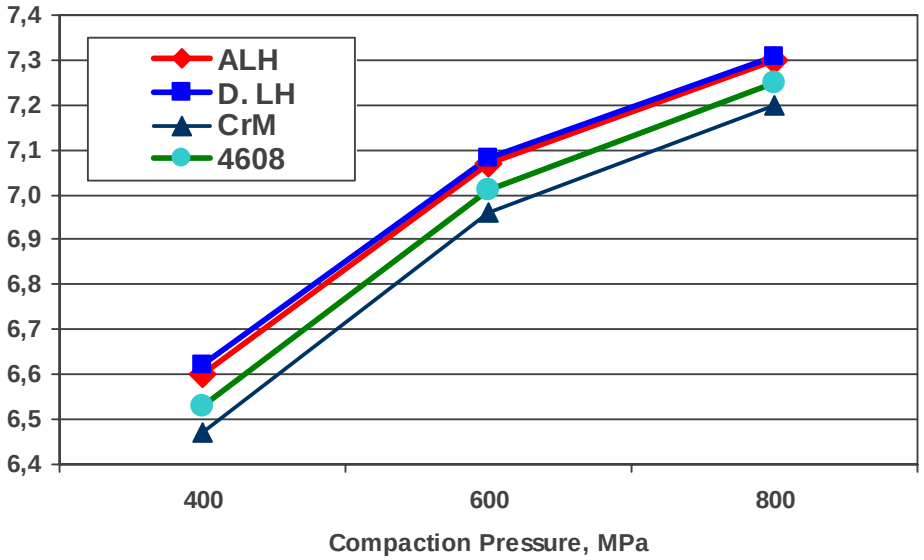


Fig. 1. Compressibility of different base powders used

Testing

After the final tempering operation, apparent hardness measurements were performed on the surface of the specimens using a Rockwell hardness tester. All of the testing was performed on the Rockwell C scale for comparison purposes. Tensile testing was performed in a MTS Sintech 20D tensile testing machine. The gauge length was 25 mm (1 inch) and the specimens were pulled at 1mm/min. Impact energy tests were carried out in a Charpy tester. Plane bending fatigue tests were carried out on un-notched compacted specimens at a fully reversed load ratio of R=-1. Endurance limit was evaluated at 2 million cycles.

Metallography

The tensile test bars were sectioned and prepared for metallographic analysis. Photomicro-graphs were taken of the structures after etching in Pikral 4% for the D. LH and etchant 3.3 for the other materials. The martensite and bainite content was determined utilizing point count analysis. This analysis technique included the porosity as a portion of the total microstructure. The reported percentages were corrected for the porosity present in the materials.

HARDENABILITY

Alloy composition, carbon content and cooling rate have a strong influence on the final microstructure of the material. Chromium and molybdenum are elements that increase hardenability and thereby suppress the pearlite formation. At lower cooling rates the microstructure will transform to mainly ferrite, pearlite and/or upper bainite. At higher cooling rates the resulting microstructure will mainly consists of martensite. To obtain homogenous microstructures after sinter hardening, it is favorable to have a homogenous distribution of the alloying elements already in the compacted part. By utilizing powders pre-alloyed with

alloyed with chromium, nickel, molybdenum, martensitic structures can be formed after sintering at 1120ºC already at moderate cooling rates, 1.5~2.5ºC/s with proper amounts of carbon.

The specimens used to investigate the hardenability were pressed to 7.0 g/cm^3 and sintered in $90N_2/10H_2$ atmosphere for 20 minutes at 1120ºC. They were then held at 1120ºC in vacuum in the dilatometer for 10 minutes before they were cooled at well defined cooling rates down to room temperature. Fractions of martensite were determined by light optical microscopy of polished and etched sample cross sections.

As all specimens were pressed to 7.0 g/cm^3, no effect of density is presented. A high density will have a positive effect on mechanical properties, but this is a direct effect of the density, not due to any change in microstructure. With a high density, the heat conductivity increases, and one could therefore expect a slightly higher cooling rate. However, the effect is usually small and difficult to detect. The microstructures obtained at 7.0g/cm^3 are therefore representative also for higher densities.

Figure 2 shows amount of martensite formed after sinter hardening with different cooling rates for D. LH and FLC-4600 (Astaloy A+2%Cu). As both materials are based on powders pre-alloyed with Mo and Ni, the hardenability behaviour is similar for the two materials. The total alloying content of FLC-4600 is somewhat higher than for D. LH, resulting in a slightly lower compressibility and higher cost compared to D. LH. At low cooling rates the structure becomes mainly bainitic, but at standard cooling rates the dominating structure is martensite, and in sinter hardening processes a completely martensitic structure can be obtained. FLC-4600 has a somewhat better response at lower cooling rates, < 0.5ºC/s, while D. LH ensures higher levels of martensite at cooling rates in the range 1.5-3ºC/s.

A high carbon content increases the martensite content at slow cooling rates for D.LH but at higher sinter hardening rates the effect is very small. High carbon levels can be used to ensure martensite also in more massive sections of specimens, where it is difficult to maintain a high cooling rate, but if this is not a problem, there should be no need to use high carbon content; 0.55% graphite could be quite sufficient for a sinter hardening operation.

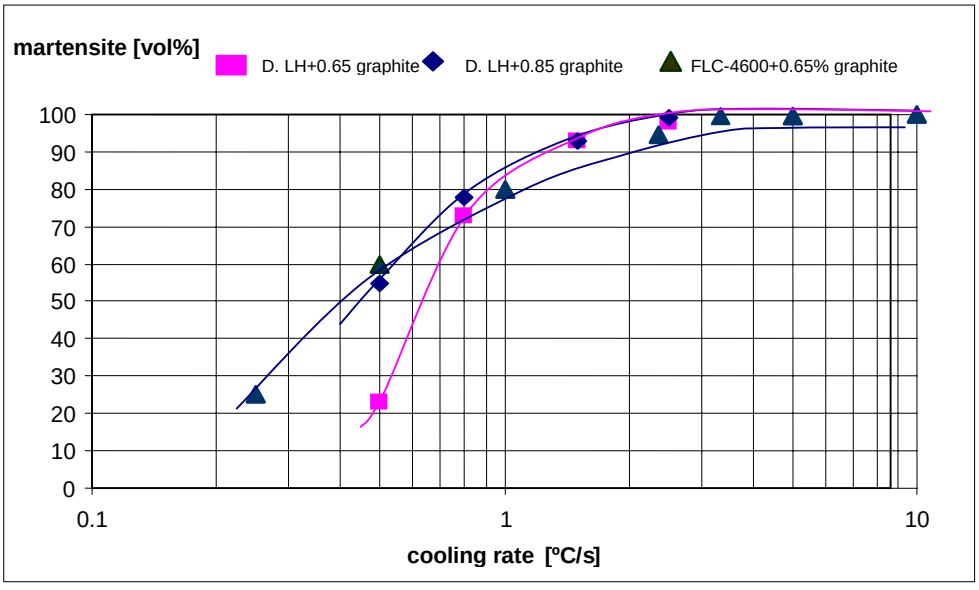
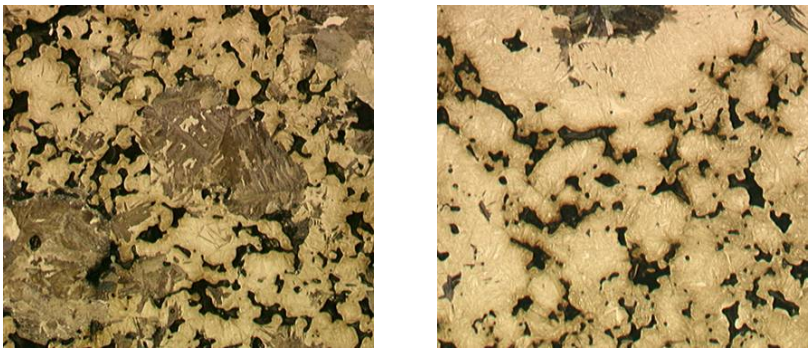


Figure 2. Martensite content vs. cooling rate for D. LH+0.65% graphite, D. LH+0.85% graphite, and FLC-4600 (Astaloy A+2%Cu)+0.65% graphite.

Figure 3 shows micrographs of etched structures in D. LH. In the micrographs the bright regions are martensite, and the dark brown regions are bainite. The image shows clearly that high levels of martensite can be obtained at moderate sinter hardening cooling rates,~1.5ºC/s.



D. LH+0.77C 0.5ºC/s D. LH+0.78C 1.5ºC/s

Figure 3.Microstructure of D. LH after sinterhardening with different cooling rates, 0.5 and1.5C/s

In Figure 4 below hardenability curves for the CrL(FL-5208) material are presented. For sinter hardening of CrL, cooling rates >2ºC/s are necessary, otherwise pearlitic/ bainitic structures are obtained. If high martensite contents are requested also at lower cooling rates, Cu can be admixed. This significantly improves the response at low cooling rates.

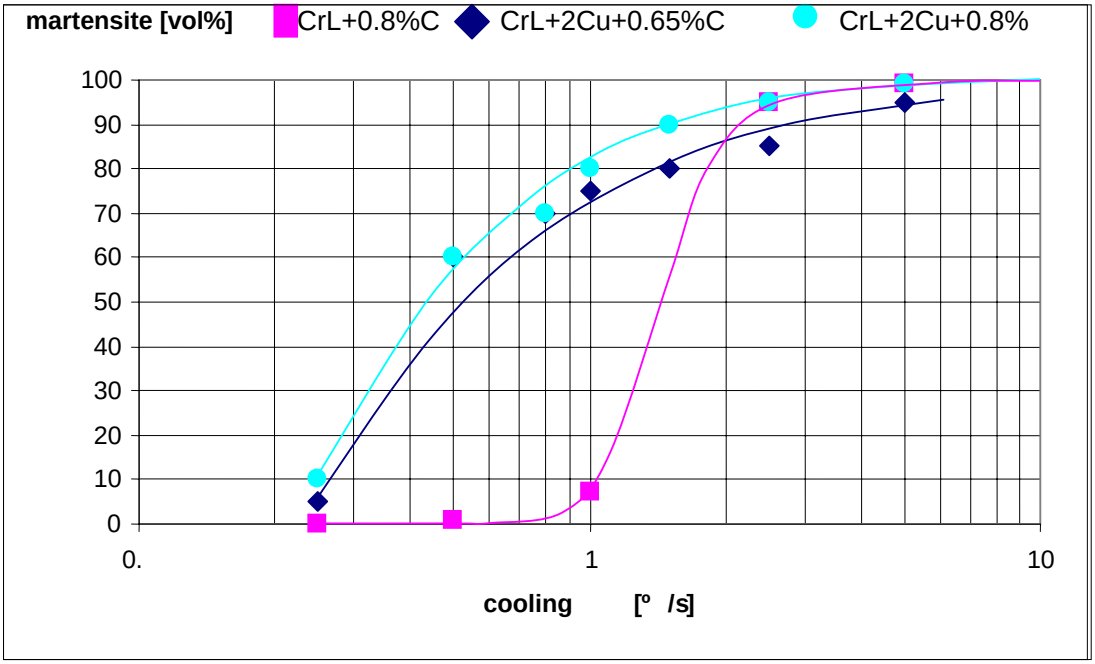


Figure 4. Martensite content vs. cooling rate for CrL+0.8C%, CrL+2%Cu+0.65% C and CrL+2%Cu+0.8%C respectively.

The more high alloyed CrM (FL-5305) shows good hardenability already at 0.4% graphite, Figure 5. At 0.5% graphite the hardenability is excellent with a stable martensitic plateau from a cooling rate of 1ºC/s. At lower cooling rates there will be some fine bainite present. This is clearly seen in Figure 6, where the slow cooled material shows a structure of fine bainitic needles in an otherwise martensitic structure. In the sinterhardened material, only small traces of bainite can be observed. The high stability in microstructure makes CrM a suitable choice

suitable choice when sinter hardening parts which contains both thin and more massive sections, and high strength is requested. If the sintering furnace can not manage chromium regarding oxygen potential the D. LH material is an equally good choice.

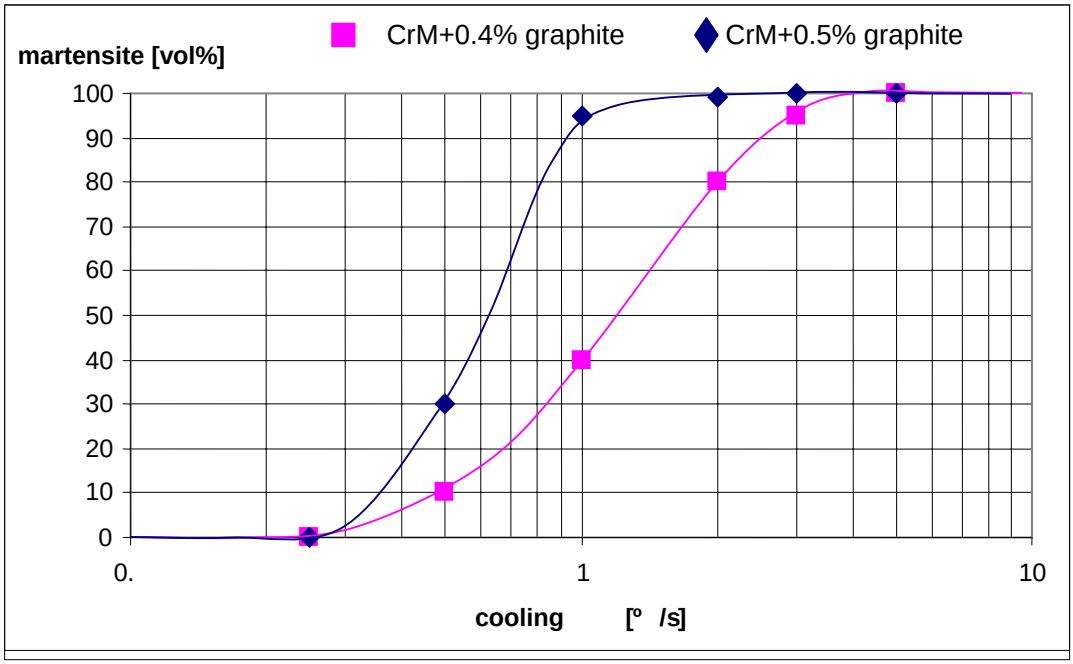


Figure 5. Martensite content vs. cooling rate for CrM+0.4% C and 0.5%C.

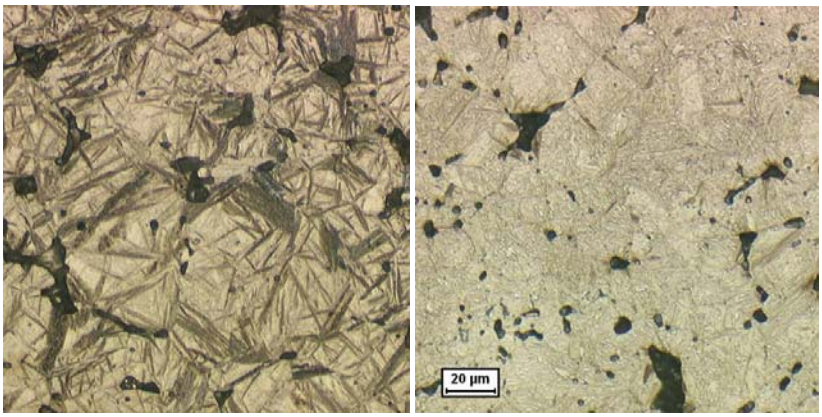


Figure 6. Microstructure of CrM after cooling at 0.5ºC/s (left) and 2ºC/s (right). Sintered carbon content is 0.45% for both materials.

RESULTS AND DISCUSSION

Hardness

A comparison of the hardness levels obtained at the two different density levels is shown in Table 3. As can be seen the hardness has increased for all materials when the density is increased from 7.00 to 7.15 g/cm^3 which is to be expected. The hardness levels obtained for the CrM material at the lower density level is the same as for the CrL material and slightly lower compared to the other materials. This is mainly due to the lower carbon content, 0.45%.

However, at the higher density level the CrM material shows comparable hardness as the D. LH and ALH materials.

Table 3. Hardness of cold compacted samples after sintering and tempering

	Density, g/cm^3	**Material**				
		CrL	**CrM**	**FLC-4608**	**D. LH**	**ALH**
Hardness, HRC	7.00	33	33	35	37	35
Hardness, HRC	7.15	34	40	37	40	41

Mechanical properties

The mechanical properties obtained after sinter hardening of the five materials are shown in Table 4. As can be seen all properties increased with increasing density.

Table 4. Mechanical properties of cold compacted samples after sintering and tempering

Material	**CrL**	**CrM**	**FLC-4608**	**D. LH**	**ALH**
Density 7.00 g/cm^3					
Tensile strength, MPa/ ksi	850 / 121	955 / 136	889 / 127	955 / 136	925 / 132
Yield strength, MPa / ksi	800/ 114	928 / 132	815 / 116	825 / 118	840 / 120
Impact energy, J / ft/lbf	16 / 10	15 / 9	16 / 10	17 / 10	17 / 10
Density 7.15 g/cm3					
Tensile strength, MPa / ksi	935 / 134	1060 / 151	940 / 135	1060 / 151	1015 / 145
Yield strength, MPa / ksi	905 / 120	1020 / 146	910 / 130	908 / 130	935 /133
Impact energy, J / ft/lbf	19 / 11	18 / 11	19 / 11	20 / 12	20 / 12

Figure 7 below shows the tensile strength obtained for all materials at the two density levels, 7.00 and 7.15 g/cm^3. As can be seen the ultimate tensile strength increased about 10% for the two Cr alloyed and the D. LH and ALH materials when density was increased from 7.00 to 7.15 g/cm^3. The CrM material and the D. LH material achieved the same tensile strength at both density levels, 955 and 1060 MPa respectively. The CrL and FLC-4608 material achieved comparable results at both density levels and about 10 % lower compared to the other three materials. It is also worth to point out that the the carbon content of the CrM material was 0.45% compared to 0.85% for the other materials and without any Cu addition.

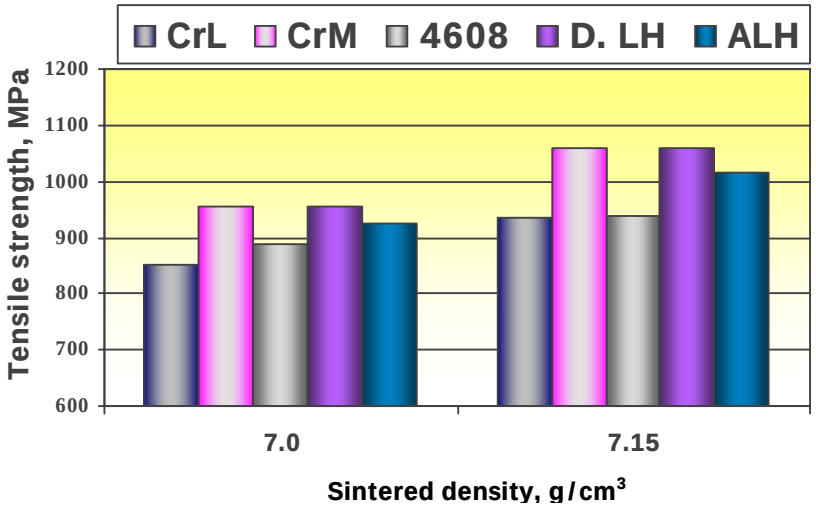


Figure 7. Ultimate tensile strength obtained for the different materials after sinterhardening at density of 7.00 and 7.15 g/cm^3.

The increase in density resulted in improvements in yield strength of about 10% for all materials. The CrM material obtained the highest yield strength at both density levels, 928 and 1020 MPa respectively. All the other materials obtained similar yield strength at the two density levels, 800 to 840 MPa at the lower level and 905 to 935 at the higher density level.

By comparing the impact energies obtained for the five materials it can be seen that the D. LH and ALH materials achieved the highest level, 17 J at the lower density and 20J at the higher density. The other three materials obtained a slightly lower impact energy at both densities.

Regarding elongation no data is included in the table but all materials achieved similar levels, about 0,3 to 0,5 % depending on the density level. The highest level was as expected obtained at the higher density level.

Plane bending fatigue strength

The positive effect of increased cooling rate after sintering at 1120°C on plane bending fatigue strength is shown in Figure 8 below. For material A the fatigue strength is increased about 25 % and for material B the increase is almost 35%. It should be pointed out that higher bending fatigue strength levels could be further increased by combining increased cooling rate with sintering at higher temperature.

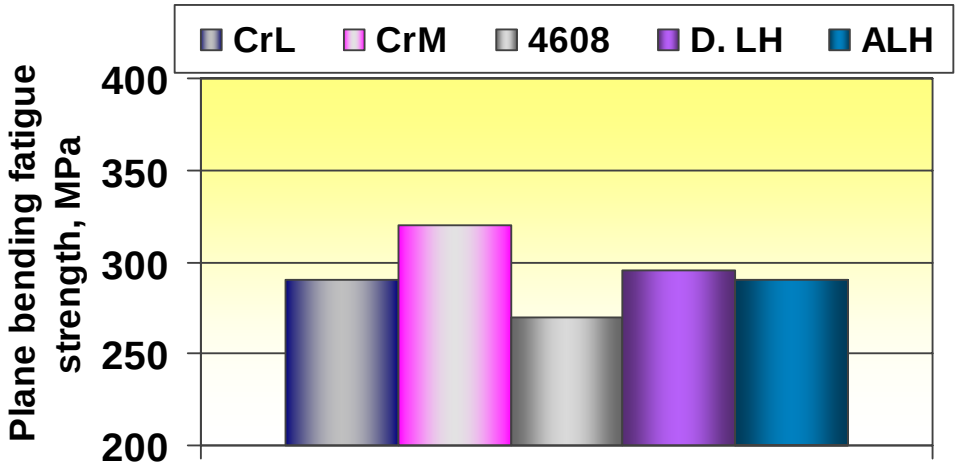


Figure 8. Bending fatigue strength obtained for the five materials after sinter hardening at 1120°C (2050°F) and at a density level of 7.00 g/cm^3

Dimensional change

The dimensional change obtained for the five materials during sintering is shown in Table 5. As can be seen the materials containing Cu all show a swelling of about 0,2-0,3 % , which is due to alloying with copper Cu which promotes growth during sintering. Regarding CrM it shows a slight shrinkage during sintering with a dimensional change close to zero.

Table 5. Dimensional change for the different materials at density 7.00 g/cm^3

Material	**CrL**	**CrM**	**FLC-4608**	**D. LH**	**ALH**
DC, %	0,23	-0,02	0,39	0,32	0,33

INFLUENCE OF SIZE AND MASS

Slugs with an outer diameter of 40 mm and varying heights, 10 , 15, 20 and 30 mm, were compacted to simulate the different sizes of components manufactured in the PM industry. In this study the D. LH mix outlined in Table 2 were compared with a mix containing 0,65%C and 1,5% Cu in order to see the influence of carbon, copper and mass effect on the resulting microstructure. The slugs of the two powder mixes were compacted to a green density of 7.00 g/cm3. Sintering was performed for 30 minutes at 1120°C in a 90 v/o N_2 / 10v/o H_2 atmosphere. The cooling rate of the furnace was set to correspond to 1.5ºC/s for the smallest slugs. The slugs were then tempered at 200°C for 1 hour in air. Apparent hardness and microstructure were then evaluated for each slug size.

The results are summarized in Table 6 below. As can be seen the D. LH material with 0.85%C obtained a hardness level exceeding 36 HRC when the mass increased from 88 g to 264 g. For the other material containing 1,5%Cu and 0,65%C the hardness decreased continuously with increasing mass. By comparing the microstructures achieved for the two materials when the mass is increasing it is clear that the amount of martensite formed has decreased significantly with increasing mass for the material with the lower amount of Cu and C. The martensite content is decreased from almost 100 % for the smallest slug to a almost completely bainitic microstructure for the largest slug. The D.LH with 0.85 %C material on the hand showed a mainly martensitic structure for all slug sizes.

Table 6. Surface hardness and vol % martensite after sinter hardening of slugs with 40 mm diameter with different heights and weights

	Slug size (O.DxH), mm	40 x 10	40 x 15	40 x 20	40 x 30
	Weight, g	88	126	176	288
Material	Eff. cooling rate, °C/s	1.5	1.3	1.2	1.0
DLH (1.5%Cu)+0.65%C	Vol. % Martensite	90-93	70-75	65-70	2-5
	Hardness, HRC	34	31	30	10
DLH+0.85%C	Vol. % Martensite	98	97	95	85
	Hardness, HRC	37	37	37	36

COMPARISON OF COST AND PERFORMANCE FOR DIFFERENT PM STEELS

The properties of PM materials depend on their density and sintering processes. The results presented in this paper show that the Cr-Mo pre-alloyed materials CrL (FL-5208) and CrM (FL-5305) on one hand and the newly developed materials D. LH and Astaloy LH on the other all provide favorable mechanical properties after sinter hardening. From Figure 9 it can be seen, that the Cr-Mo pre-alloyed steels and the new D. LH and Astaloy LH materials can achieve comparable or superior tensile strength using less alloying content than traditional Fe-Cu, Fe-Cu-Ni, and Fe-Ni-Cu-Mo steels.

The cost effectiveness of the two pre-alloyed Cr materials, CrL and CrM, relative to other sinter hardening grades makes these materials very competitive for new high performance sinter hardening applications.

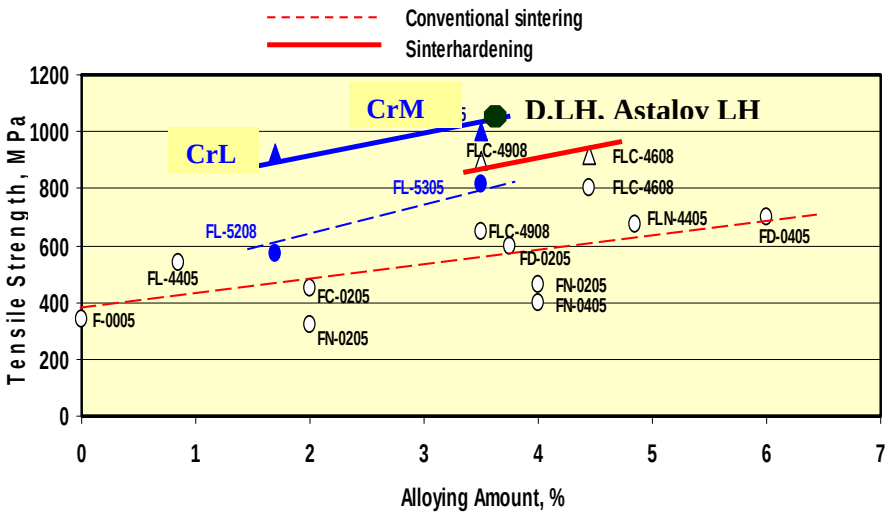


Figure 9. Comparison of strength for CrL (FL-5208, CrM (FL-5305), D. LH and Astaloy LH with traditional P/M materials

CONCLUSIONS

The effect of post sintering cooling rate on microstructure and the resulting mechanical properties of the two fully pre-alloyed Cr materials, Astaloy CrL (FL-5208) with 2% Cu addition and Astaloy CrM (FL-5305) and the two new cost effective pre-alloyed Ni /Mo powders Astaloy LH and the diffusion alloyed material D. LH can be summarised as follows:

D. LH and Astaloy LH are two new cost effective sinter hardening solutions with high compressibility that can be sintered in any sintering atmosphere, including endothermic gas.

Astaloy CrM (FL-5305) and Astaloy CrL (FL-5208) are the most cost effective sinter hardening solutions when the sintering process can handle sintering of Cr in a proper way.

Accelerated cooling resulted in increased tensile and yield strength values. At a cooling rate of 2ºC/s tensile strength levels exceeding 1050 MPa were obtained for both the D. LH material and for Astaloy CrM.

REFERENCES

1. Akpan, E., L' F@sperance, G., Roy, L., "Case Histories with Sinter Hardening Low Alloy Steel Powder", *Advances in Powder Metallurgy & Particulate Materials* - 1993, Vol. 4, p. 289, Metal Powder lndustries Federation, Princeton, NJ. .

2. Höganäs Handbook for Sintered Components. &. Metallography. 1999, p 197.

3. U. Engström, ”Evaluation of sinterhardening of different PM materials”, *Advances in Powder metallurgy & Particulate Materials*, 2000, p 5-147 to 5-157