Chromium pre-alloyed PM steels suitable for high performance applications

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ABSTRACT

Steel powders pre-alloyed with chromium are successfully used in PM components for high performance applications. Good mechanical properties are obtained after conventional sintering and the properties can be further improved by the use of high temperature sintering and/or sinter-hardening. Case hardening operations may also be applied for applications where the PM component surfaces are highly loaded. This paper presents dynamic as well as static mechanical properties obtained for different Cr-Mo pre-alloyed powder grades after various process routes. Compaction pressure, sintering temperature and cooling rate have been varied and case hardening has been applied. Correlations between microstructures and mechanical properties are described for the different materials. The benefits of using case hardening are highlighted, which is demonstrated by the excellent fatigue properties obtained.

INTRODUCTION

Chromium is an interesting alloying element, not only because it provides excellent possibilities to reach high performances in sintered components, but also because of the low cost and good recycling possibilities. Chromium has since long been used in conventional steels because of its high hardenability, good resistance to temper softening, and relatively low cost. The use in powder metallurgy has, however, been limited, mainly because of the high affinity for oxygen. Nevertheless, with correct processing, the oxidation problem can be sufficiently reduced, and in recent years two water atomized steel grades with chromium as prealloyed element have been introduced; Astaloy CrM with 3% chromium and 0.5% molybdenum, and Astaloy CrL, with 1.5 % chromium and 0.2% molybdenum [1,2].

The prealloyed route offers some advantages over the additive route. Successful sintering at conventional temperature (1120ºC/2048ºF) becomes possible in nitrogen-based atmospheres. The prealloyed route also makes high temperature sintering possible without disregarding the demands in PM industry for tighter tolerances in the production of high performance parts.

All stages in the processing of PM parts have impact on final properties. Already in the compaction stage some of the final mechanical properties of the sintered components are established. During sintering the temperature affects neck formation, pore roundness, austenite grain size, and oxide reduction. Final properties are determined by the austenite transformation, which is controlled by the cooling rate. Materials prealloyed with chromium and molybdenum offers a wide spectrum of microstructures. By optimising carbon content and process route, final microstructure can be controlled, and chromium pre-alloyed powders can be used to produce PM parts suitable for high performance applications.

This paper summarises properties achieved by different compaction techniques, sintering temperatures, and cooling rates. Also several different microstructures in the Fe-Cr-Mo-C system are presented.

EXPERIMENTAL

Materials

As base powders Astaloy CrL and Astaloy CrM were used. The composition of the powders can be found in Table 1.

Table 1. Composition of the base powders.

Material	Cr (%)	Mo (%)	Fe
Astaloy CrL	1.5	0.2	Balance
Astaloy CrM	3.0	0.5	Balance

To investigate the effect of different processing on the base powders, different series of powder mixes were prepared. These mixes are presented in Table 2.

Table 2. Powder mixes used in the investigation.

Material	Base powder	Graphite (%)	Lubricant
A1	CrL	0.8	0.7% A-wax
A2	CrL	0.8	0.6 % Densmix
A3	CrM	0.5	0.7% A-wax
A4	CrM	0.5	0.6 % Densmix
B1	CrM	0.55	0.8% A-wax
B2	CrM	0.65	0.8% A-wax
B3	CrM	0.75	0.8% A-wax
B4	CrM	0.85	0.8% A-wax
C	CrM	0.45	0.6 % Densmix
D	CrL	0.8	0.8% A-wax

Compaction

Tensile testing bars, ISO 2740, and modified ISO 3928 fatigue bars with chamfered edges were used in the investigation. Also cylindrical dilatometer specimens with a diameter of 6 mm and a height of 12 mm were used.
The A materials were compacted using conventional cold compaction and warm compaction at 400-800 MPa (58-116*10^3 psi). Also high velocity compaction was used [3]. The resulting green densities were in the range of 7.0-7.5 g/cm^3.
The B materials were compacted using conventional cold compaction to a density of 7.0 g/cm^3.
Material C was warm compacted to a density of 7.15g/cm^3.
Material D was cold compacted to dilatometer specimens with a density of 7.0 g/cm^3.

Sintering and heat treatment

The A materials were both conventionally sintered at 1120ºC (2048ºF), and high temperature sintered at 1250ºC (2282ºF). The sintering was in both cases performed for 30 minutes at temperature in 90/10% N_2/H_2 atmosphere. Two different cooling rates were applied: 0.5-1ºC/s (standard cooling) and

2-3ºC/s (sinter-hardening). The sinter-hardened specimens were tempered in air at 200ºC (392°F) for 60 min.
The B materials were high temperature sintered at 1250ºC (2282ºF) for 60 minutes in 90/10% N_2/H_2 atmosphere. Both standard cooling and sinter-hardening were applied. All materials, except material B1 cooled at 0.8ºC/s were tempered in air at 200ºC (392°F) for 60 min.
Material C was sintered at 1300ºC (2372ºF) in a laboratory vacuum furnace for 30 min. Subsequent cooling to 1050ºC (1922ºF) was performed, after which the specimens were vacuum carburized with acetylene gas for 8 min. After carburizing, the specimens were cooled in N_2 atmosphere at a pressure of 2 bar. The specimens were moved to another vacuum furnace, and a second heat treatment was performed at 950ºC (1742ºF) for 15 min. Subsequent cooling was performed in N_2 atmosphere at 7 bar pressure. The specimens were tempered in air at 200ºC (392ºF) for 60 min. Bulk carbon content after processing was approx. 0.3%.
Before analysis in the dilatometer, the material D specimens were sintered at 1120ºC for 20 min in 90/10% N_2/H_2 atmosphere. After sintering, a hole with a diameter of 3 mm was drilled in the specimens in order to facilitate cooling in the dilatometer runs.
In the dilatometer, the specimens were heated to 1120ºC (2048ºF) in vacuum, where a holding time of 10 min was applied for austenitizing. The specimens were then fast cooled down to transformation temperature, at which they were held for 15 minutes. Several transformation temperatures between 700 and 250ºC (1292 and 482ºF) were applied, after which the specimens were quenched with N_2.
For all heat-treatments where cooling rate has been specified, this refers to the temperature range 800-300ºC (1742-572ºF).

Fatigue testing

The fatigue properties were tested in plane bending. S-N curves were determined at a frequency of about 25 Hz and a load ratio R=-1. Run outs were considered at 2 million cycles. Fatigue strength limit was determined with stair case method according to MPIF standard no. 56.

RESULTS AND DISCUSSION

Different processing

Compaction

PM materials generally have lower properties than traditional wrought materials, due to internal porosity. Final mechanical properties can, however, be improved by pressing to high green densities. This can be achieved by simply increasing the compaction pressure, or by using some more advanced technique than cold compaction [4]. By using warm compaction, the compaction process becomes more effective, and higher densities can be reached without increasing compaction pressure [5]. By using the high velocity compaction technique, the green density can be increased further.
Results from pressing of Astaloy CrL and CrM with different techniques are presented in Figure 1. As can be seen, the green density can be significantly improved by using warm compaction instead of cold compaction. By using the high velocity compaction technique, the green density can be taken to a further level, and an increase of more than 0.4g/cm^3 can be achieved.

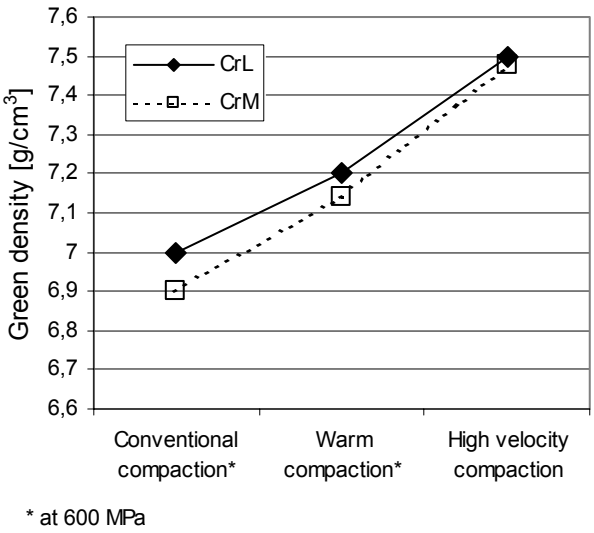


Figure 1. Green densities obtained for Astaloy CrL and Astaloy CrM with different compaction techniques.

The effect of density on mechanical properties was investigated for Astaloy CrM with 0.4% C, and for Astaloy CrL with 0.75% C (material A1-4 in Table 2). The results are visualised in Figure 2, which shows the effect of density on elongation and tensile strength. The positive effect from density is clearly seen. An increase from 6.9 to 7.3g/cm^3 in sintered density leads to, approximately, a 50% increase in elongation, and a 25% increase in tensile strength.

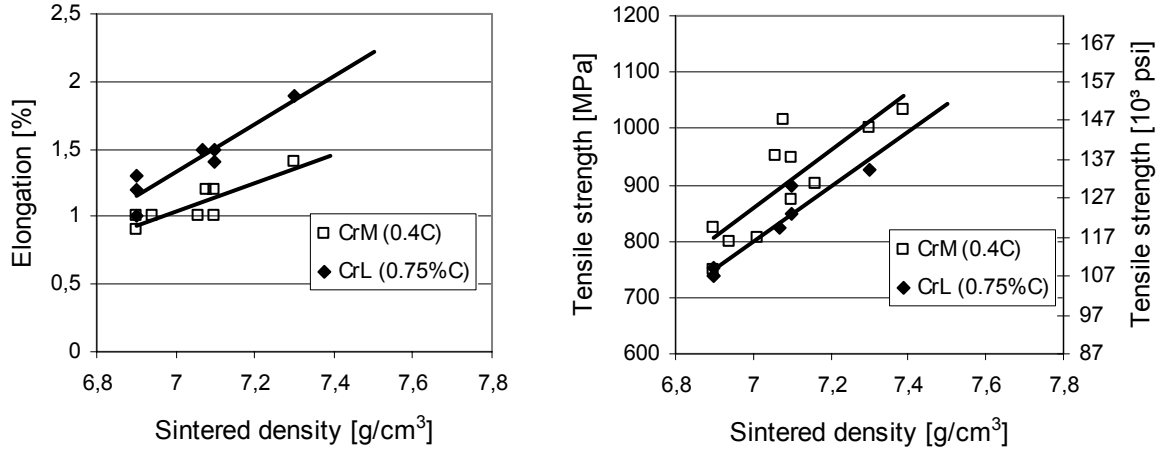


Figure 2. Mechanical properties vs. sintered density for Astaloy CrL and Astaloy CrM; elongation (left), and tensile strength (right).

High temperature sintering

Conventionally, sintering is performed at 1120ºC (2048ºF), with a cooling rate of approx. 0.5-1ºC/s. With proper atmosphere control, this sintering temperature is sufficient for a pre-alloyed chromium-containing material [6]. However, higher sintering temperature can be beneficial. When the sintering temperature is increased, diffusion and sintering activity is also increased, resulting in stronger sintering necks, more rounded pores, and more effective oxide reduction. These factors increase the

mechanical properties of a high temperature sintered material. Not only the strength is increased, but also, as a result of the more favourable pore structure, the ductility is increased.
In Figure 3 the positive effect of increased sintering temperature on tensile strength and elongation is visualized. As sintering temperature is raised from 1120ºC (2048ºF) to 1250ºC (2282ºF), the elongation is significantly increased for both conventionally cooled and sinter-hardened materials.

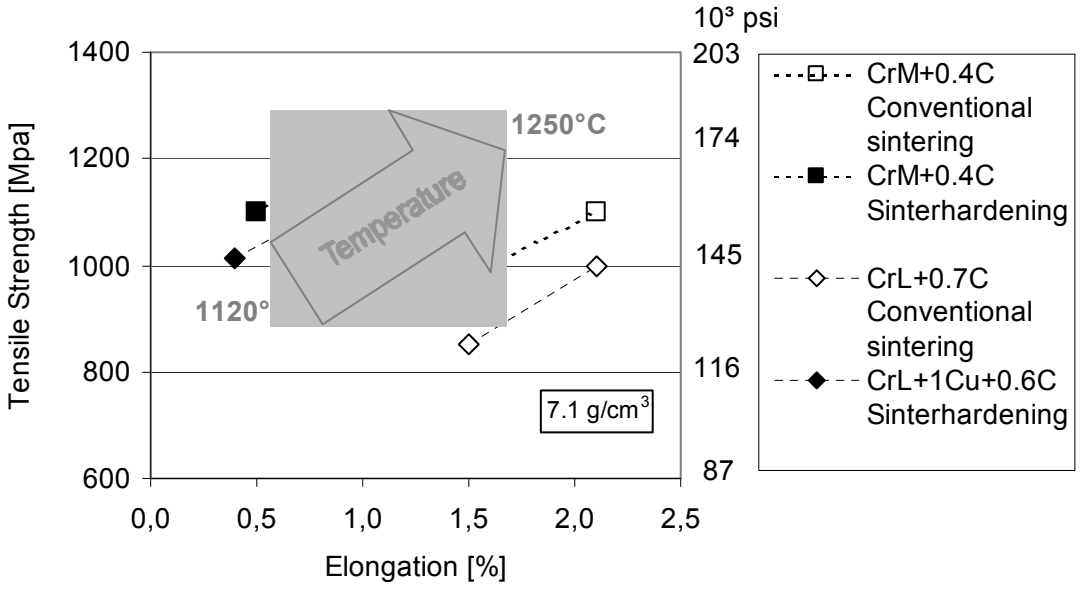


Figure 3. Effect of sintering temperature on tensile strength and elongation.

The sintering temperature also affects the final microstructure. A high sintering temperature results in larger austenite grain size. This might have some effect on austenite transformation upon cooling. When transformation to pearlite or bainite takes place, the main nucleation sites for these structures are grain boundaries. With a large austenite grain size the grain boundary area is reduced, i.e. the available nucleation sites are reduced. Nucleation should then be slower, and in a continuous cooling process, it could be shifted towards lower temperature. In special cases a high sintering temperature might therefore slightly increase hardenability.

Sinter-hardening

The cooling rate in the temperature range 850ºC ⇒ 300ºC highly affects the final microstructure. With a low cooling rate, the austenite will transform at high temperature, and the transformation products will be mainly ferrite, pearlite and/or upper bainite. If a high cooling rate is applied, there will not be sufficient time at high temperature for transformation. Instead the transformation takes place at lower temperature, and the products will be stronger microstructures, mainly martensite. This is of course also highly dependent on chemical composition. Chromium and molybdenum are elements that suppress the pearlite formation, i.e. they shift the pearlite transformation to longer times and increase hardenability. By adding chromium and molybdenum, together with carbon, in proper amounts to a steel, martensitic structures can be formed at moderate cooling rates (~2.5ºC/s), so-called sinter-hardening.
To obtain homogenous microstructures after sintering, it is important to have a homogenous distribution of the alloying elements. With chromium as a prealloyed element, the response for sinter-hardening is therefore better than for a material with chromium admixed, and sinter-hardening of a chromium prealloyed material is possible after sintering at 1120ºC.
Figure 4 shows Tensile strength for chromium prealloyed materials with a sintered density of 7.3 g/cm^3 (Astaloy CrL and Astaloy CrM), subjected to sinter-hardening and/or high temperature

sintering. It is clear that the processing increases the strength of the material. Lowest strength is obtained for conventional sintering temperature (1120ºC/2048ºF) and conventional cooling rate (0.5-1ºC/s). The use of high temperature sintering or sinter-hardening increases tensile strength with more than 20%, to over 1100 MPa ($160*10^3$ psi) for Astaloy CrL, and to over 1200 MPa ($174*10^3$ psi) for Astaloy CrM. If high temperature sintering is combined with sinter hardening, even higher strength is obtained. Astaloy CrL can reach a strength of 1200 MPa ($174*10^3$ psi), and Astaloy CrM a strength of 1400 MPa ($203*10^3$ psi).
Figure 5 shows the elongation for the same materials as in Figure 4. For this property the variations with processing are more complex. High temperature sintering increases the elongation, while sinter-hardening decreases the same. The reason is found in the microstructure, where the larger amount of martensite in the sinter-hardened materials decreases the elongation. By combining high temperature sintering and sinter-hardening, the elongation can be kept at a good level at the same time as the strength is significantly increased.

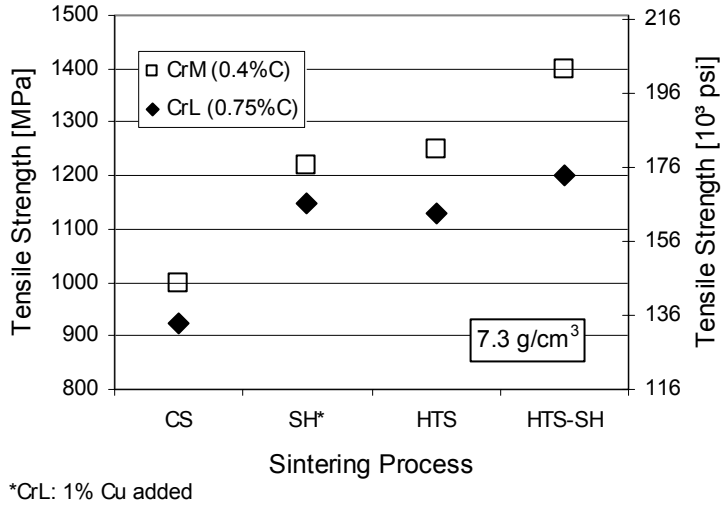


Figure 4. Tensile strength for Astaloy CrL and Astaloy CrM after different sintering processes. CS: conventional sintering, SH: sinter-hardening, HTS: high temperature sintering.

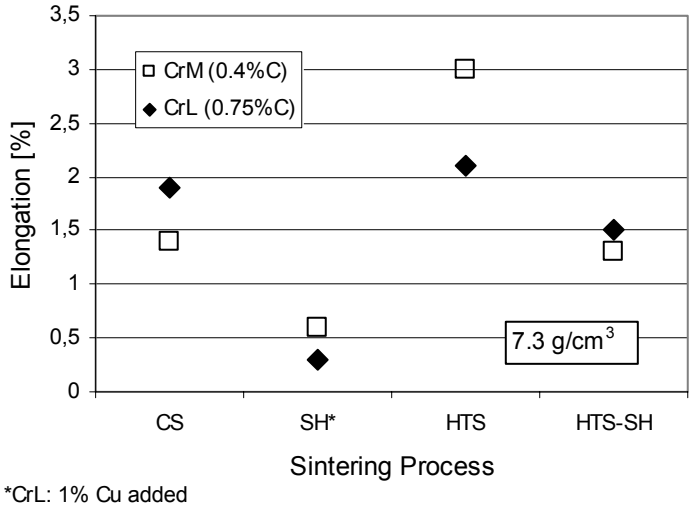


Figure 5. Elongation for Astaloy CrL and Astaloy CrM after different sintering processes. CS: conventional sintering, SH: sinter-hardening, HTS: high temperature sintering.

The carbon content highly affects a material's response to sinter-hardening. High temperature sintering (1250ºC/2282ºF) of Astaloy CrM with different carbon contents (materials B1-4, Table 2) was carried out with both conventional cooling and sinter hardening to investigate this effect. The influence is visualised in Figure 6, where yield strength, hardness, and elongation is presented, and in Figure 7, which shows fatigue properties for conventionally cooled material.
A large increase in strength is found for the conventionally cooled material when the carbon content is increased from 0.4 to 0.5%. Also the hardness increases drastically in this carbon interval. The explanation for this can be found in Figure 8 and Figure 9. At a carbon content of 0.4% and a cooling rate of 0.8ºC/s the resulting microstructure will be mainly bainite with some martensite. With an increase in carbon to 0.5%, the microstructure will change towards a mainly martensitic structure with some bainite.
For the sinter-hardened material the effect from increased carbon content is not as pronounced because of the high martensite content created already at 0.4% carbon.
As the carbon content is increased further, the hardness stabilizes, and is approximately the same for both conventionally cooled and sinter-hardened materials (500 HV10). The strength is somewhat higher for sinter-hardened materials than for conventionally cooled materials at high carbon content, but above 0.5%C it decreases for both cooling rates. The reason for the decrease in strength is the more brittle martensite formed at high carbon content, and also the presence of retained austenite.
The behaviour of the elongation is the inverse of the hardness, with somewhat higher values for the conventionally cooled materials.
In Figure 7 the fatigue strength is shown. It is important to notice that while yield strength and hardness decrease for high carbon contents, fatigue strength continues to increase, i.e. there is no correlation between optimum carbon content for static properties and for fatigue properties.

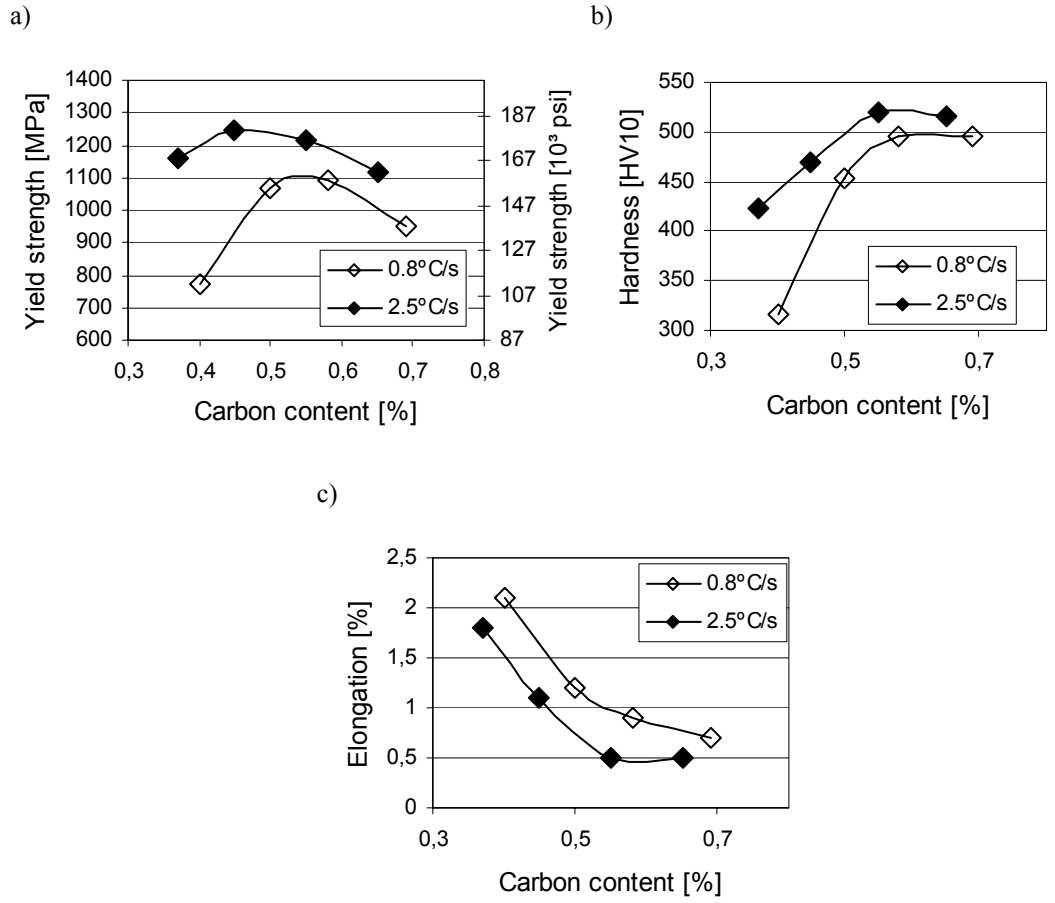


Figure 6. Effect of carbon content on mechanical properties for high temperature sintered Astaloy CrM. a) yield strength, b) hardness, c) elongation.

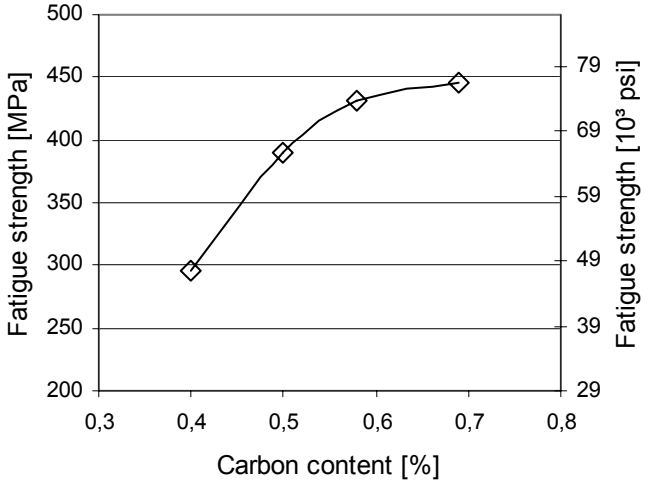


Figure 7. Influence of carbon content on fatigue strength for high temperature sintered Astaloy CrM.

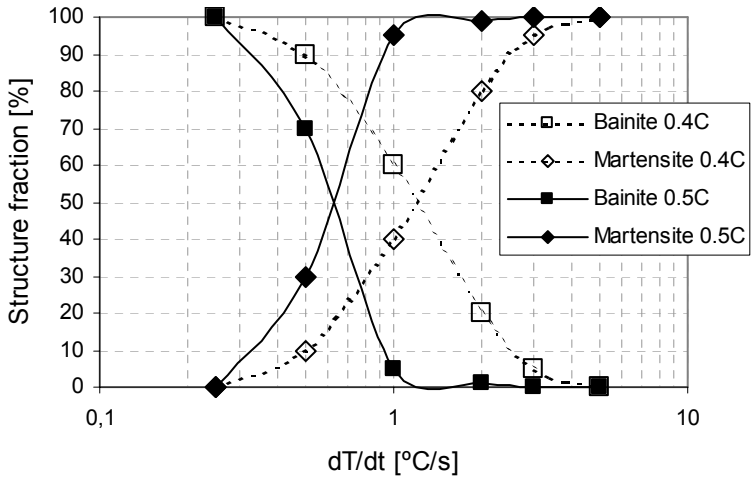


Figure 8. Structure amount vs. cooling rate for Astaloy CrM with 0.4 and 0.5% C.

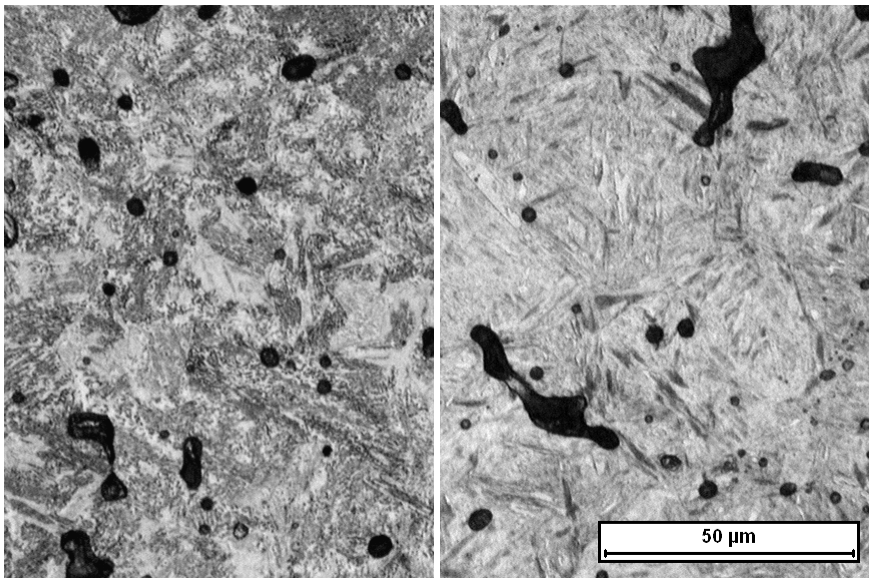


Figure 9. Astaloy CrM with 0.39% C, cooled at 0.8ºC/s (left), and 2.5ºC/s (right)

Compared to Astaloy CrM, the variations in structure with cooling rate for Astaloy CrL are more complex. As an example Figure 10 shows the variations with cooling rate for Astaloy CrL with 0.46 and 0.74% C. Due to the overall lower alloying content, pearlite is formed at low cooling rates. As cooling rate increases towards 1ºC/s, the structure changes towards bainite. At cooling rates above 2ºC/s martensite starts to form, but for low carbon contents bainite dominates the structure even at high cooling rates.

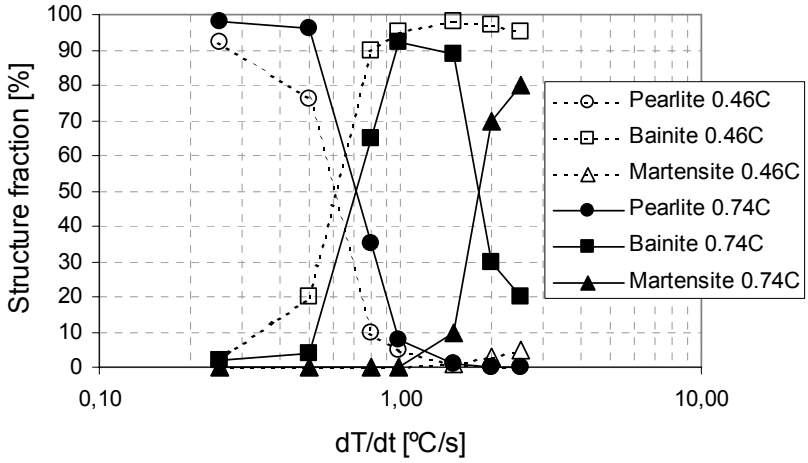


Figure 10. Structure amount vs. cooling rate for Astaloy CrL with 0.46 and 0.74% C.

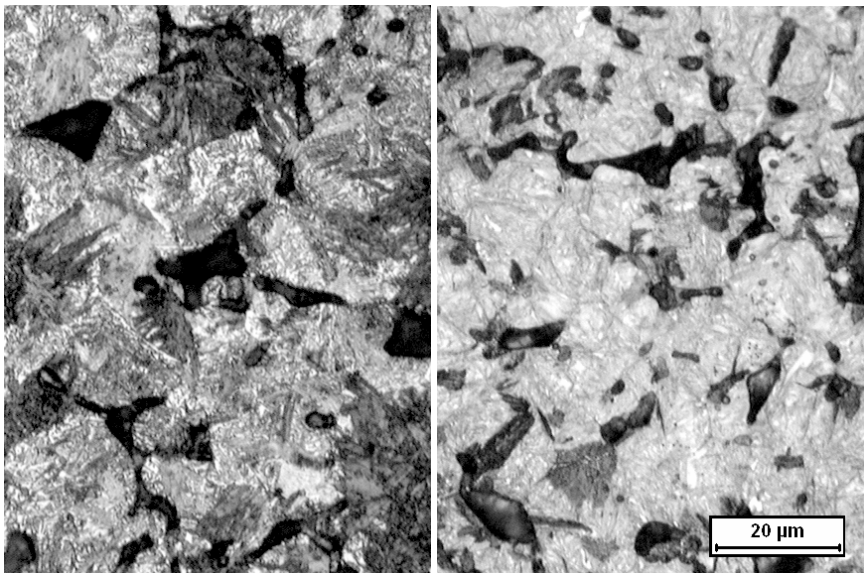


Figure 11. Astaloy CrL with 0.74% C cooled at 0.8ºC/s (left), and 2.5ºC/s (right).

Case hardening

Case hardening is a process used for components when a hard case and a tough core are desirable. Table 3 shows the resulting properties for material C, which was high temperature sintered and vacuum carburized. The carburization has created a martensitic structure at the surface, while the interior consists mainly of bainite. The result of the process is a high surface hardness and high fatigue strength (527 Mpa/76*10^3 psi). The hardness profile can be seen in Figure 12. Case hardening depth, defined as the distance from the surface where the hardness drops below 550 Vickers units, is approx 0.4mm.

Table 3. Properties for vacuum carburized Astaloy CrM.

C as-sint.[%]	Sint. Density [g/cm3]	Hardness [HV10]	Fatigue strength [MPa/10^3 psi]	
			σA,50%	St. dev
0.3	7.29	507	527/76.6	<5/0.7

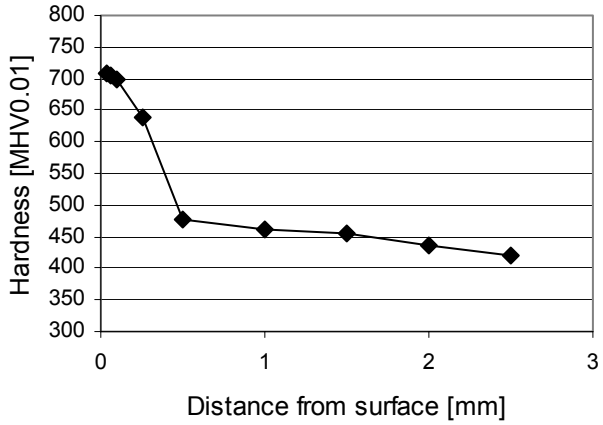


Figure 12. Microhardness profile for vacuum carburized Astaloy CrM.

Isothermal transformation of Astaloy CrL

The alloying content of Astaloy CrM suppresses the pearlite formation at conventional cooling rates, and makes the material suitable when bainitic or martensitic structures are desired. Astaloy CrL, on the other hand, with the comparably low alloying content offers a wider range of microstructures. By optimising carbon content and cooling rate, final dominating structures can be selected (see Figure 10).

When a material is continuously cooled, the resulting microstructure originates from transformations over a certain temperature range. By performing isothermal transformations, different structures inside this temperature range can be refined. To investigate the temperature dependence on microstructure for Astaloy CrL with approximately 0.73% C (material D in Table 2) the material was austenitized, and then fast-cooled to a certain temperature for isothermal transformation. Figures 13-16 show the resulting microstructures in SEM micrographs of etched specimens.

At 700ºC (1292ºF) pearlite is formed (Figure 13). The structure varies with regions of fine and coarse pearlite.

At 650ºC (1202ºF) the pearlite is finer than at 700ºC (Figure 13). The material does, however, still contain regions of rather coarse pearlite. For the regions of fine pearlite light optical microscopy is not sufficient, but SEM is necessary to resolve the lamellas.

As temperature is decreased, the structure becomes increasingly finer. At 600ºC (1112ºF) and 550ºC (1022ºF) a very fine pearlite is formed (Figure 14). This pearlite starts to lose the lamellar appearance, and it looks rather spiky [7, 8, 9]. It is no longer possible to resolve the structure using LOM.

When the transformation is performed at 525ºC (977ºF), bainite starts to form (Figure 15). The structure is a mix of coarse upper bainite, fine pearlite, and some martensite. As can be seen in the micrograph, the bainite is much coarser than the pearlite.

At 450ºC (842ºF) complete bainite reaction takes place (Figure 15). The structure is predominantly upper bainite, but some lower bainite has started to form. The upper bainite becomes finer with decreasing transformation temperature.

At 300ºC (572ºF) almost complete transformation to lower bainite takes place (Figure 16). Only small amounts of martensite are found in the materials. The presence of martensite is probably due to an incomplete bainite reaction.

When transformation takes place at 250ºC (482ºF) only small amounts of bainite can be found, and the structure is mostly martensitic (Figure 16).

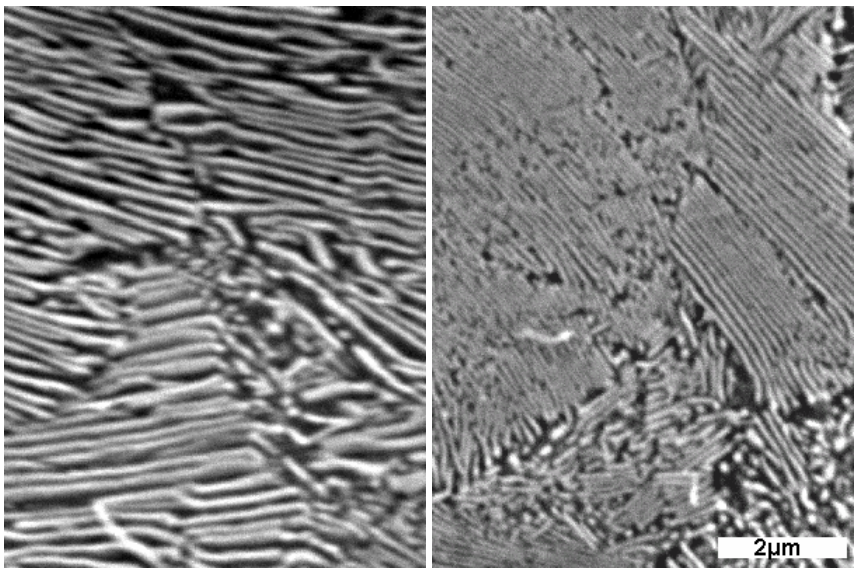


Figure 13. SEM micrograph of Astaloy CrL with 0.73% C transformed at 700ºC/1292ºF (left), and with 0.73% C transformed at 650ºC/1202ºF (right).

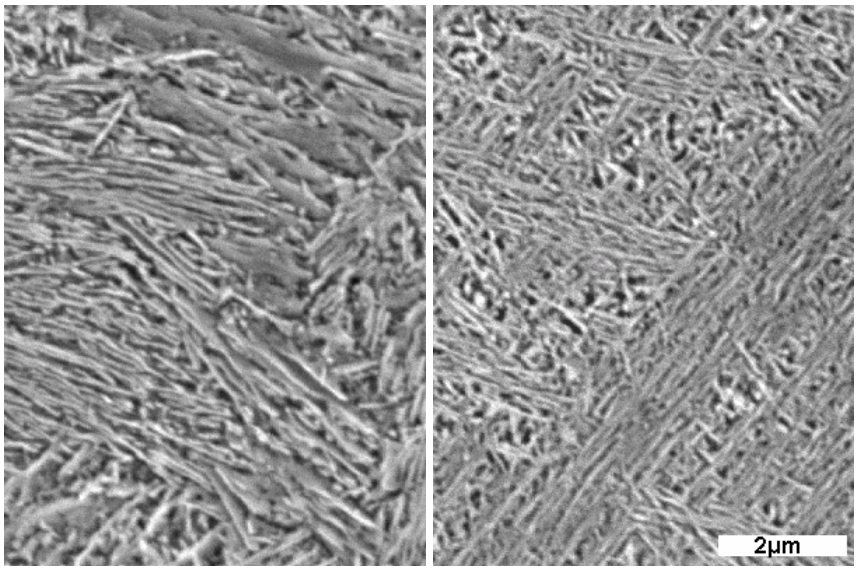


Figure 14. SEM micrograph of Astaloy CrL with 0.71% C transformed at 600ºC/1112ºF (left), and with 0.73% C transformed at 550ºC/1022ºF (right).

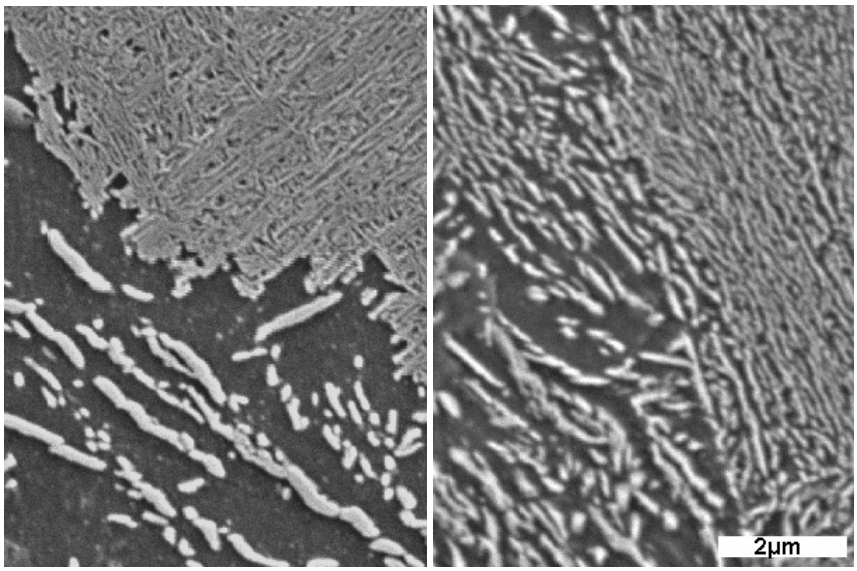


Figure 15. SEM micrograph of Astaloy CrL with 0.74% C transformed at 525ºC/977ºF (left), and with 0.73% C transformed at 450ºC/842ºF (right).

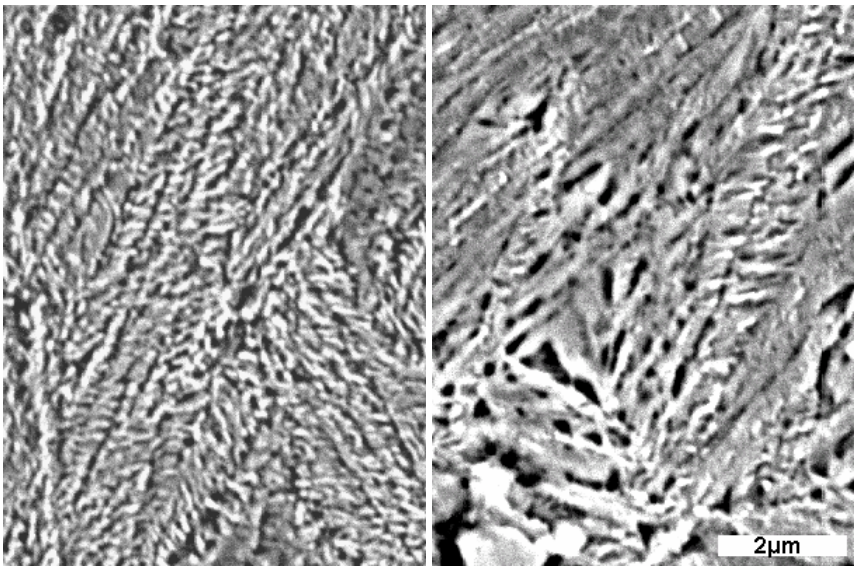


Figure 16. SEM micrograph of Astaloy CrL with 0.71% C transformed at 300ºC/572ºF (left), and with 0.72% C transformed at 250ºC/482ºF (right).

To get an indication of the mechanical properties of the different structures obtained by isothermal transformations, microhardness measurements were performed. The results are presented in Figure 17. From the graph it is clear that the fine pearlite is harder than the coarse upper bainite, but when the bainitic structure shifts from upper to lower bainite, the hardness is significantly increased.

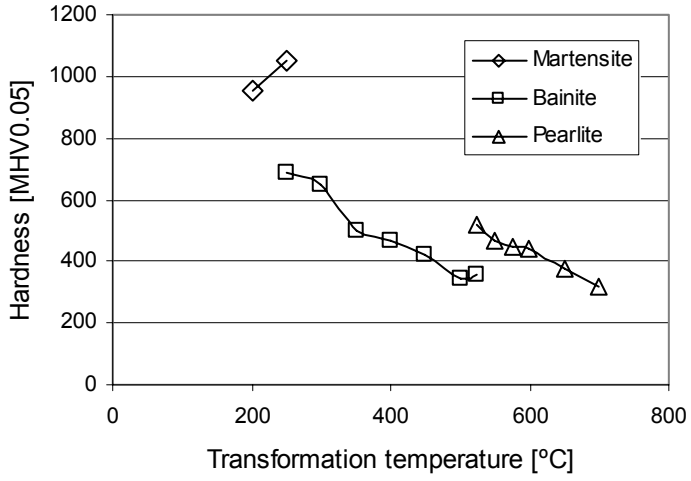


Figure 17. Microhardness vs. transformation temperature for Astaloy CrL with 0.73%C.

CONCLUSIONS

Materials prealloyed with Cr and Mo possess good hardenability, and give excellent response to sinter-hardening operations. Fully martensitic structures can be achieved at a cooling rate of 2.5ºC/s.

Optimum yield strength of 1100 Mpa ($159*10^3$ psi) for Astaloy CrM is found at 0.58% C for conventionally cooled materials, and 1250 Mpa ($181*10^3$ psi) at 0.45%C for sinter-hardened material.

Plane bending fatigue increases with increasing carbon content, and for Astaloy CrM at 0.7%C and 7.2g/cm^3, a strength of 450 MPa (65*10^3 psi) is reached.

Vacuum carburizing can be used to increase fatigue strength. Warm compaction of Astaloy CrM, in combination with high temperature sintering and vacuum carburizing gives a plane bending fatigue strength of 530 MPa (76*10^3 psi) at 0.3%C and 7.3 g/cm^3.

With high temperature sintering of Cr-Mo prealloyed materials, both strength and ductility increase.

In Astaloy CrL a very fine pearlite can be formed, which is superior to upper bainite in microhardness.

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