PRE-ALLOYED CHROMIUM MATERIALS FOR HIGHLY FATIGUE LOADED PM PARTS.

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ABSTRACT

The use of PM materials in highly loaded components is continuously expanding. Fatigue properties on such parts are becoming more and more important. Pre-alloyed chromium materials enables cost efficient productions of highly fatigue loaded PM parts. In this paper fatigue performance of pre-alloyed chromium material sintered at 1120°C(2050°F), 1250°C(2280°F) and 1280°C(2340°F) are presented. Cold compaction to 7.0 – 7.1 g/cm^3 and warm compaction to 7.3 g/cm^3 are used. The materials are processed to high performance microstructures obtained directly in as sintered state after normal cooling. Shot peening has been shown to be a very efficient process route on Chromium pre-alloyed materials with fine pearlite or tempered martensite microstructures. The results of the Chromium pre-alloyed materials are compared to diffusion alloyed high performance material D.HP1. Dynamic data are presented and correlations between microstructures and mechanical properties are stated.

INTRODUCTION

Two features of PM steel are related to high fatigue performance, namely microstructure and density. Microstructure is the most important factor. Martensite is the most fatigue resisting microstructure in PM and ferrite the least. Density also contributes to the fatigue performance but it must be pointed out that very high fatigue performance can be reached also at densities around 7.0 g/cm^3 provided the microstructure is optimized. For example a shift of microstructure from pearlite in a Fe-Cu-C material to diffusion alloyed D.HP1+0.8%C material with heterogeneous microstructure characterized by upper bainite, martensite and Ni-rich austenite typically increases the bending fatigue limit by 50% from 220 to 330 MPa at density 7.1 g/cm^3. Increased density raises the fatigue limit of the high performance microstructures about 3-10% per 0.1 g/cm^3 corresponding to an increase of 12-40% when the density is raised from 7.0 to 7.4 g/cm^3.

Heterogeneous distributions are obtained from powder mixes or diffusion-alloyed materials where alloying elements with slow diffusion rates in steels at the sintering temperature are utilized. Copper-

carbon PM steel is one example where Cu-rich solid solution hardened pearlite is found along the sinter necks and pearlite and ferrite in the cores of the base powders. Copper and Nickel diffusion alloyed PM steels in combination with either pre-alloyed or diffusion-alloyed Molybdenum utilizes the slow diffusion rate of Nickel in iron and transient liquid phase sintering from Copper. Copper diffuses very quickly along the sinter necks and forms martensite together with Molybdenum. Ni-rich islands of austenite surrounded by martensite play an active role to arrest fatigue cracks [1]. In this way martensite is created exactly where it is needed, namely along the sinter necks.

PM steels made from pre-alloyed powders with fatigue resistant microstructures is another way to get high performance PM steels. One specifically successful group of materials is those based on Chromium/Molybdenum pre-alloyed powders. The microstructures of these materials are characterized by upper bainite, fine pearlite, lower bainite and martensite depending on Chromium and Molybdenum content, addition of graphite and cooling rate.

This paper focuses on the fatigue behavior of two Chromium pre-alloyed PM steels in as sintered states and after secondary operations. Comparison is made with a diffusion alloyed PM steel.

The fatigue performance of PM steels can be improved by secondary operations. Through hardening, case hardening or shot peening (or a combination) are process routes to get highest possible fatigue resistance of a component. These secondary operations have the largest impact on components with notches and stress concentrations. Through hardening is performed to get martensite. Case hardening combines two positive characteristics, namely a hard surface that shows high wear and contact stress resistance with excellent fatigue endurance limit from the combination of surface martensite and compressive residual stresses. Shot peening is normally performed in order to utilize the beneficial influence of compressive residual stresses in the surface. Pores open to the surface are weak points in PM materials that are at least partly neutralized by introduction of surface compression residual stresses.

Two examples of secondary operations on Chromium materials, namely shot peening of as sintered material and the combination of through hardening, tempering and shot peening on notched fatigue specimens, will be presented in this paper.

EXPERIMENTAL

Materials and microstructures

The three materials included in this study are shown in Table I.

Table I. Two pre-alloyed and one diffusion-alloyed base powders included in the study. D.HP1 is diffusion-alloyed with Ni and Cu and pre-alloyed with Mo.

Material	Ni [%]	Cu [%]	Mo [%]	Cr [%]
Astaloy CrL			0.2	1.5
Astaloy CrM			0.5	3.0
D.HP1	4.0	2.0	1.5	

Detailed information on process parameters, density and carbon levels will be given for each material in the result section. Low and high temperature sintered materials are included in the study. Results are presented for all three materials sintered at 1120°C(2050°F) in 90%N_2/10%H_2 with an oxygen potential of $5 \cdot 10^{-18}$ corresponding to a dew point of -30°C(-22°F). Results are also presented for AstaloyCrL sintered at 1250°C(2280°F) and 1280°C(2340°F) and AstaloyCrM sintered at 1250°C(2280°F).

Specimens and fatigue tests

Fatigue tests on un-notched specimens were performed using 5 mm ISO3928 samples with chamfered edges see Figure 1. The tests were made in four-point plane bending at load ratio R=-1. The staircase method was used with 10 – 18 samples in the staircase and 2 million cycles as run-out limit. Evaluation of the staircase (50% probability fatigue limit, standard deviation and calculated 90% probability limit) was made according to the MPIF 56 standard. Test frequency was 27 – 30 Hz. Tests on samples after secondary operations were made with fewer numbers.

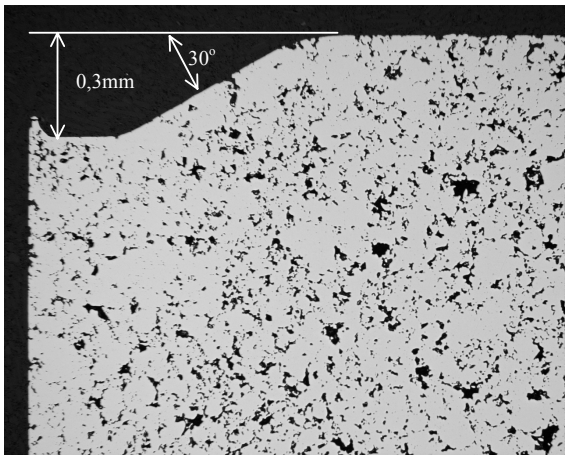


Figure 1. Modified ISO 3928 test specimen with chamfered edges.

RESULTS

AstaloyCrL

The microstructure of AstaloyCrL with sintered carbon below 0.6% and cooling rate about 0.8°C/s (1.4°F/s) is upper bainite. Increased carbon above 0.74% changes the microstructure to fine pearlite.

Process parameters and results from plane bending fatigue tests are shown in Table II. The S-N diagrams are shown in Figure 2.

Table II. Plane bending fatigue performance of AstaloyCrL with cooling rate about 0.8C/sec.

Powder	Sintering	Density [g/cm^3]	Carbon As-Sint [%]	$\sigma_{A,50\%}$ [MPa]	Std.Dev [MPa]	$\sigma_{A,90\%}$ [MPa]
AstaloyCrL	1120°C, 30min, 90/10 N_2/H_2, 0.8°C/s	7.17	0.60	244	7	234
		7.16	0.80	267	5	260

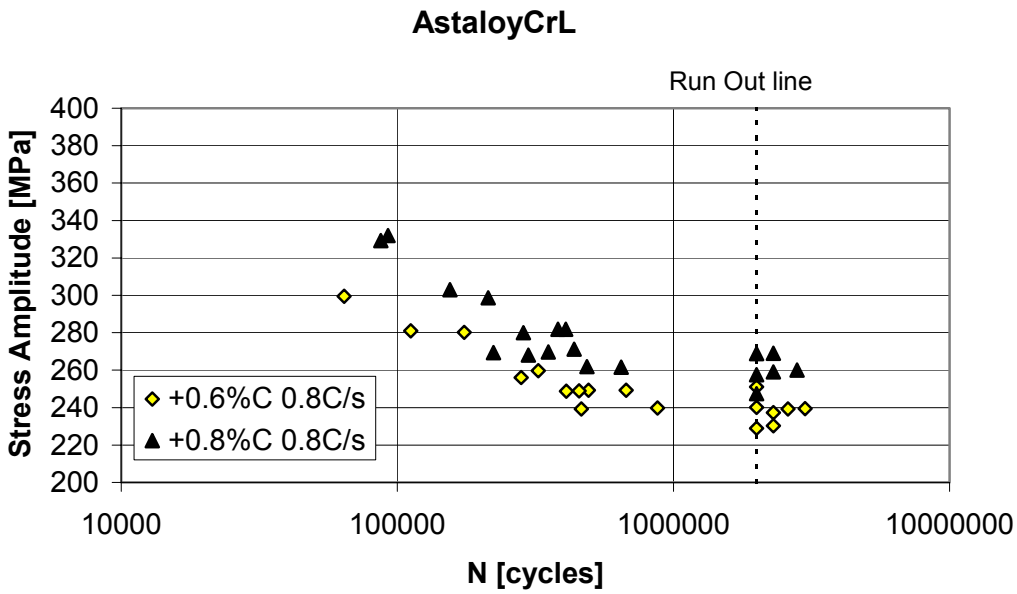


Figure 2. S-N diagrams for Astaloy CrL with sintered carbon 0.6 and 0.8 % C at density 7.17 and 7.16 g/cm^3 respectively. The fatigue limits are 244 and 267 MPa with standard deviations 7 and 5 MPa. All results on or to the right of the run-out line are run outs at 2 million cycles.

Table III shows influence of compaction pressure and carbon level for cold compacted AstaloyCrL. All materials were sintered at 1120°C(2050°F) for 30 min. in 90/10 N_2/H_2.

Table III. Summary of plane bending fatigue performance of AstaloyCrL at two compaction pressures and two levels of additional graphite. Std.dev. <5 MPa indicates that the scatter is small and the MPIF standard 56 evaluation of standard deviation cannot be applied. The standard deviation is in this case estimated to half of the step in the staircase.

Material	Graphite C-UF4 [%]	Compacting Pressure [MPa]	Density As-sint [g/cm³]	Carbon As-sint [%]	$\sigma_{A,50\%}$ [MPa]	Std.Dev [MPa]	$\sigma_{A,90\%}$ [MPa]
AstaloyCrL 1120°C, 30min, 90/10 N_2/H_2 0.8°C/s	0,6	600	6,94	0,56	224	11.6	205
	0,8	600	6,93	0,75	233	9.5	218
	0,6	800	7,13	0,55	236	8.5	222
	0,8	800	7,09	0,74	252	<5	>244

Influence of sintering temperature on the fatigue performance is shown in Table IV. The corresponding S-N diagram is shown in Figure 3. The microstructure is characterized by fine pearlite.

Table IV. Process parameters and plane bending fatigue limit for high temperature sintered Astaloy CrL. Sintered for 30 min in 90/10 N_2/H_2, followed by cooling rate 0.8°C/s(1.4°F/s).

Powder	Sint. Temp. [°C]	Compacting pressure [MPa]	Density As-Sint. [g/cm^3]	Carbon As-Sint. [%]	$\sigma_{A,50\%}$ [MPa]	Std.Dev [MPa]	$\sigma_{A,90\%}$ [MPa]
AstaloyCrL	1250	600	7.02	0.74	290	18	264

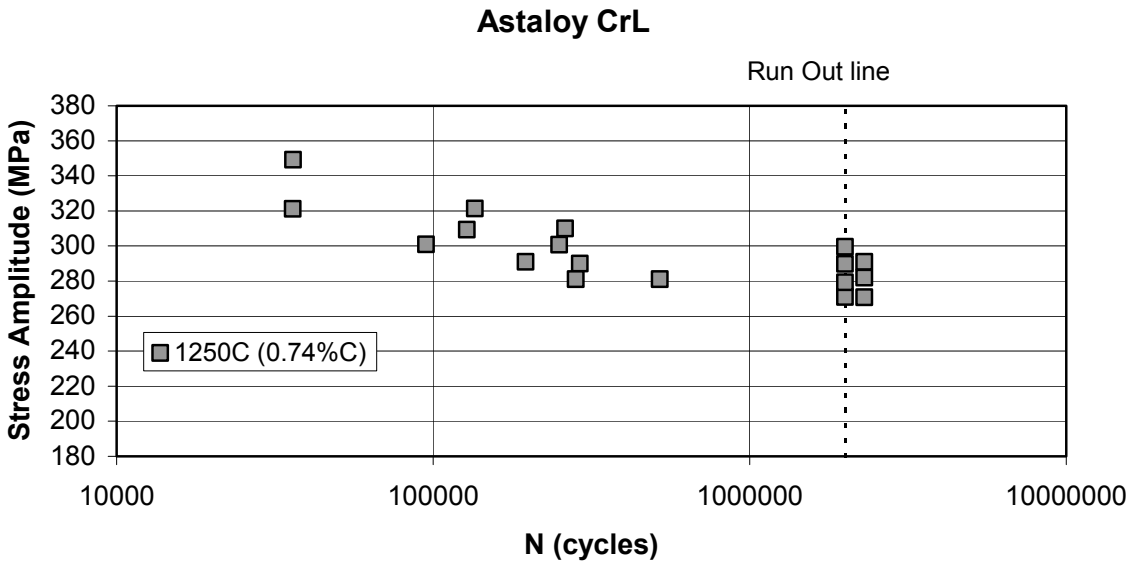


Figure 3. S-N diagram for AstaloyCrL sintered at 1250°C(2280°C). All results on or to the right of the run-out line are run outs at 2 million cycles.

AstaloyCrM

Microstructure analysis of 1120°C(2050°F) sintered AstaloyCrM materials and cooling rate 0.8°C/s (1.4°F/s) and with sintered carbon levels between 0.32% and 0.49% show a dense upper bainitic microstructure. Dense upper bainite has the same characteristics as regular upper bainite, i.e. an irregular mix of ferrite and cementite. The differences are the smaller distances between carbides and sizes of the carbides. Increased carbon to 0.56% changes the microstructure to a mixture of lower bainite and martensite.

Results from AstaloyCrM with 0.35%C with dense upper bainite and 0.56%C with 60%martensite and 40% lower bainite are shown in Table V. S-N diagrams from the fatigue tests for both carbon levels are shown in Figure 4.

Table V. Process parameters and plane bending fatigue limits for two Astaloy CrM materials sintered at 1120°C(2050°F).

Powder	Sintering	Density As-Sint [g/cm^3]	Carbon As-Sint [%]	$\sigma_{A,50\%}$ [MPa]	Std.Dev [MPa]	$\sigma_{A,90\%}$ [MPa]
AstaloyCrM	1120°C, 30min, 90/10 N_2/H_2 0.8°C/s	7.06	0.35	284	7.0	274
		7.04	0.56	316	8.4	300

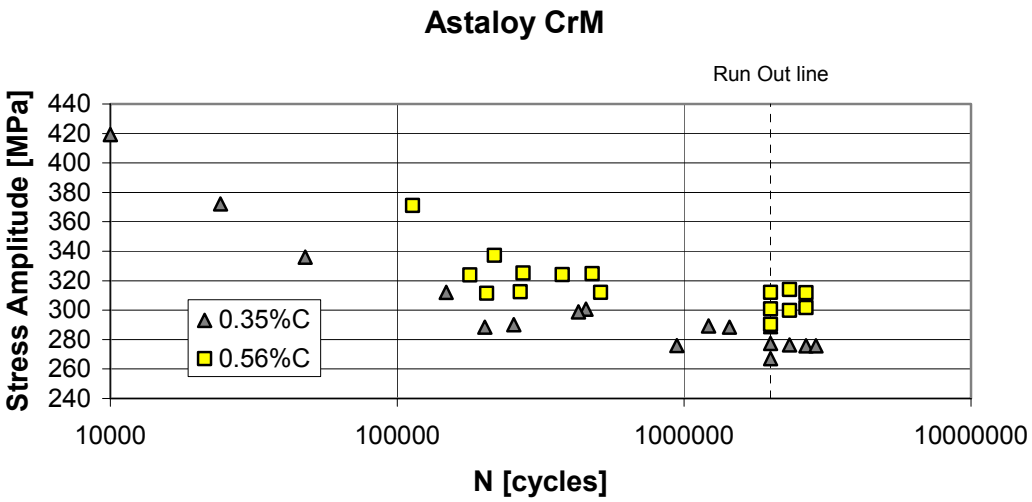


Figure 4. S-N diagrams for Astaloy CrM with 0.35%C and 0.56%C. The fatigue limits are 278 and 316 MPa respectively. All results on or to the right of the run-out line are run outs at 2 million cycles.

Results from 1250°C(2280°F) sintered AstaloyCrM with sintered carbon between 0.4 and 0.68% are shown in Table VI. S-N diagrams for carbon levels 0.40% and 0.58% are shown in Figure 5. High temperature sintered materials were sintered again at 1120°C(2050°F) in belt furnace in order to obtain the same cooling rate for all materials (0.8°C/s (1.4°F/s)).

Table VI. Process parameters and plane bending fatigue limits for two Astaloy CrM materials sintered at 1250°C(2280°F) and cooling rate 0.8°C/s (1.4°F/s).

Powder	**Sintering**	**Density As-Sint [g/cm^3]**	**Carbon As-Sint [%]**	**$\sigma_{A,50\%}$ [MPa]**	**Std.Dev [MPa]**	**$\sigma_{A,90\%}$ [MPa]**
AstaloyCrM	1250°C, 30 min, 90/10 N_2/H_2 + 1120°C, 30min, 90/10 N_2/H_2 0.8°C/s	7.17	0.40	296	19	268
		7.15	0.50	390	14	369
		7.15	0.58	432	26	392
		7.16	0.69	445	26	405

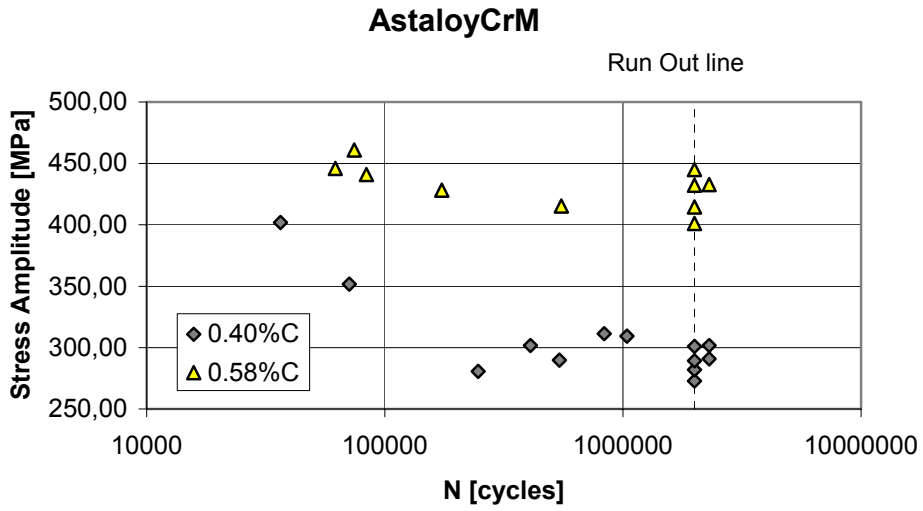


Figure 5. S-N diagrams for 1250°C(2280°F) sintered Astaloy CrM with 0.40%C and 0.58%C. The fatigue limits are 296 and 432 MPa respectively. All results on or to the right of the run-out line are run outs at 2 million cycles.

D.HP1

Results from D.HP1 with addition of 0.6 and 0.8%graphite sintered at 1120°C(2050°F) are shown in Table VII.

Table VII. Plane bending fatigue performance and standard deviation for D.HP1 with cooling rate about 0.8C/sec

Powder	Sintering	Density [g/cm^3]	Carbon As-Sint [%]	$\sigma_{A,50\%}$ [MPa	Std.Dev [MPa]	$\sigma_{A,90\%}$ [MPa
D.HP1	1120°C, 30min, 90/10 N_2/H_2 0.8°C/s	7.13	0.65	295	22.5	261
		7.13	0.85	330	<5	>322

The S-N diagrams of D.HP1 at the two carbon levels are shown in Figure 6.

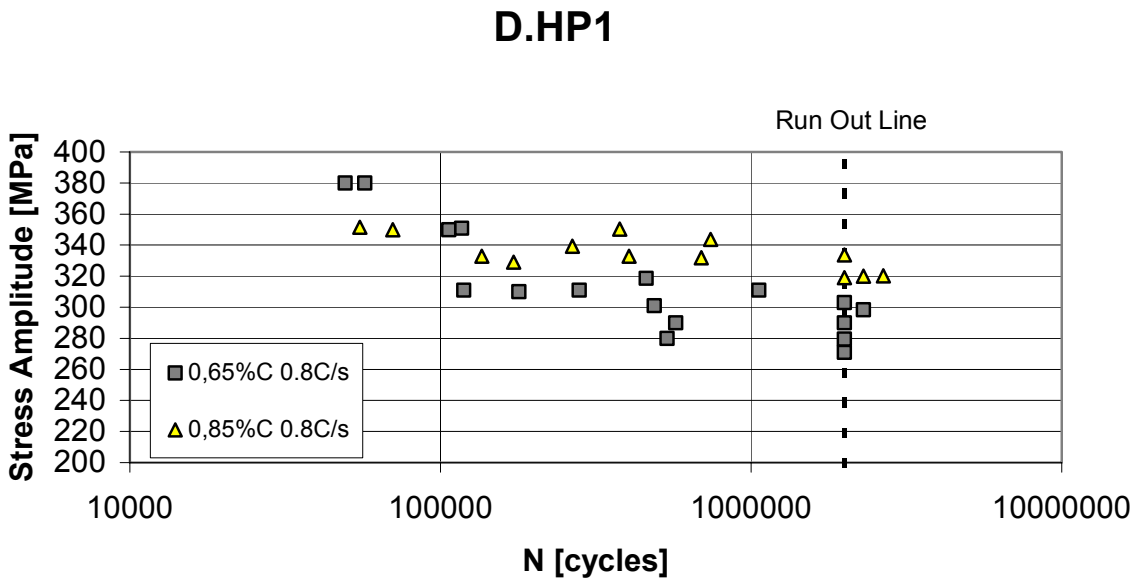


Figure 6. S-N diagrams for D.HP1 with 0.65% and 0.85%C. Sintered at 1120°C(2050°F) for 30 min in 90/10 N_2/H_2. Sintered densities 7.13 g/cm^3. The fatigue limits are 280 and 330 MPa respectively. All results on or to the right of the run-out line are run outs at 2 million cycles.

Shot peening

Influence of shot peening and the combination of heat treatment and shot peening has been investigated for materials based on Astaloy CrL using 3 mm edge-notched specimens (Figure 8). The notch is included in the press tool and no machining was performed. The stress concentration factor in bending is obtained by FEM to K_t=1.38. The materials were sintered at 1280°C(2340°F) for 30 min in H2.

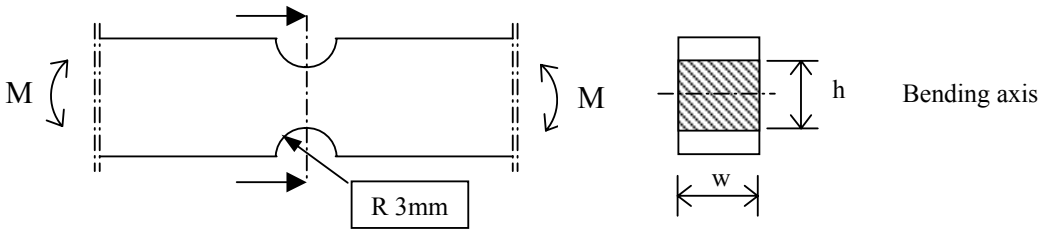


Figure 7. Geometry of tested notch bar. Minimum cross-section is 7x7mm with chamfered edges (not shown here) according to Figure 1.

Nominal stress is calculated according to:

$\sigma_{nom} = \frac{M}{\frac{w \cdot h^2}{6}} \cdot \alpha$, where α is the correction factor for influence of chamfered edges. The correction factor is calculated to $\alpha = 1.04$

Maximum theoretical stress is calculated from nominal stress and the theoretical stress concentration factor:

$\sigma_{\max} = K_t \cdot \sigma_{nom}$.

Shot peening was performed on sintered specimens based on AstaloyCrL in the as-sintered condition with microstructure characterized by fine pearlite. The performances have been estimated from the S-N curves obtained from tests with 10 samples. Results are shown in Table VIII. Shot peening increases the fatigue performance from 235 to 420 MPa nominal bending stress corresponding to +79%.

Table VIII. Estimated plane bending fatigue performance of as sintered and as-sintered plus shot peened samples. Cooling rate is 0.8°C/s (1.4°F/s).

Powder	**Carbon As-Sint [%]**	**Density As-Sint [g/cm^3]**	**Secondary operation Shot peening**	**Nominal stress σ_{nom} [MPa]**	**Theoretical max stress σ_{max} [MPa]**
AstaloyCrL	0.70	7.3	NO	235	324
			YES	420	580

Shot peening was also performed on through hardened and 250°C(480°F) /1hour vacuum tempered Astaloy CrL. The fatigue limit of shot peened samples was estimated to 490MPa from seven samples. Five of them were tested at nominal stress level 490MPa (three run-outs and two broken samples after 1.3 and 1.9 million cycles respectively) and two at 470MPa. (run-outs). The test system did not allow tests at higher stresses. The performance of not shot peened samples has been estimated from the S-N curve with 10 samples. Shot peening increases the fatigue performance from 285 to 490 MPa nominal bending stress corresponding to +73%.

Table IX. Estimated plane bending fatigue performance of through hardened tempered and shot peened samples.

Powder	**Carbon As-Sint [%]**	**Density As-Sint [g/cm^3]**	**Secondary operations**			**σ_{nom} [MPa]**	**σ_{max} [MPa]**
			Through hardening	**Tempering 250°C 1h**	**Shot peening**		
AstaloyCrL	0.50	7.3	YES	YES	NO	285	389
	0.50	7.3	YES	YES	YES	490	676

DISCUSSION

Chromium/Molybdenum pre-alloyed PM steels at densities around 7.1 g/cm^3 exhibit as sintered plane bending fatigue performance in the interval 220 MPa to 430 MPa depending on the microstructure. Carbon content and cooling rate are important factors for microstructure. All as sintered materials included in this study have the same cooling rate, about 0.8C/sec. This is achieved by using the same sintering furnace for all 1120°C(2050°F) sintered materials. A batch furnace is used for all 1250°C(2280°F) sintered materials. These materials have been re-sintered in the 1120°C(2050°F) belt furnace in order to get the same cooling rate as 1120°C(2050°F) sintered materials. The total time above 1120°C(2050°F) in the 1250°C(2280°F) batch furnace is about 3 hours. It is considered that one extra

sintering cycle for 30 minutes at 1120°C(2050°F) in this case will have a minor influence only on the mechanical performance.

Results from fatigue tests on AstaloyCrL are shown in Figure 8. The diagram shows one low carbon (0.54% to 0.60%) and one high carbon group (0.74% to 0.80%) with microstructures characterized by upper bainite and fine pearlite respectively. Mean values in each group are 235 and 252 MPa corresponding to an increase in fatigue performance of about 7% when the microstructure is shifted from upper bainite to fine pearlite. The diagram also show a fatigue limit increase from 224 to 244MPa in the low carbon group when density is increased from 6.94 to 7.17 g/cm^3 or 5%/0.1 g/cm^3. The same value is found for the high carbon group.

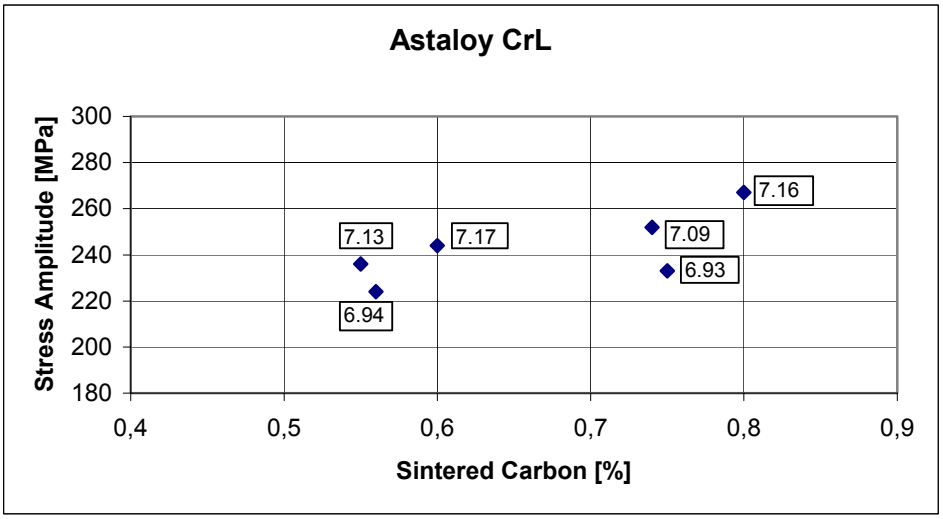


Figure 8. AstaloyCrL. Summary of data for relation between sintered carbon level and plane bending fatigue performance. Densities are included for each data point. Microstructures: Low carbon (0.55 – 0.6%): upper bainite. High carbon (0.74 – 0.8%): fine pearlite.

High temperature sintered AstaloyCrL with 0.74%C presented in Table IV show a fatigue limit of 290 MPa at density 7.02 g/cm^3. The fatigue limit for low temperature sintered AstaloyCrL with high carbon is estimated to 240MPa from Figure 8. High temperature sintering raises the fatigue of AstaloyCrL with high carbon about 20% in this case.

Results for AstaloyCrM at different carbon levels obtained after cooling rate 0.8°C/s (1.4°F/s) are assembled in Fig. 9. Increased carbon in AstaloyCrM shows a shift from dense upper bainite (DUB) at carbon <0.5% to a mixture of low temperature bainite (LB) and martensite (M) in the interval 0.5 – 0.57 % C and martensite at carbon ≥0.58 %. The diagram includes data for 1120°C(2050°F) sintered AstaloyCrM with densities around 7.05 g/cm^3 and 1250°C(2280°F) sintered with densities around 7.15 g/cm^3. The figure shows 6 materials sintered at 1120°C(2050°F). Two of them were included in Table V and Fig. 4. The performances increases with density and values obtained from materials with different sintering temperatures cannot be directly compared. However, the result obtained for dense upper bainite microstructure with fatigue limit close to 300MPa for low and high temperature sintered AstaloyCrM indicates that the influence of sintering temperature in this case is small.

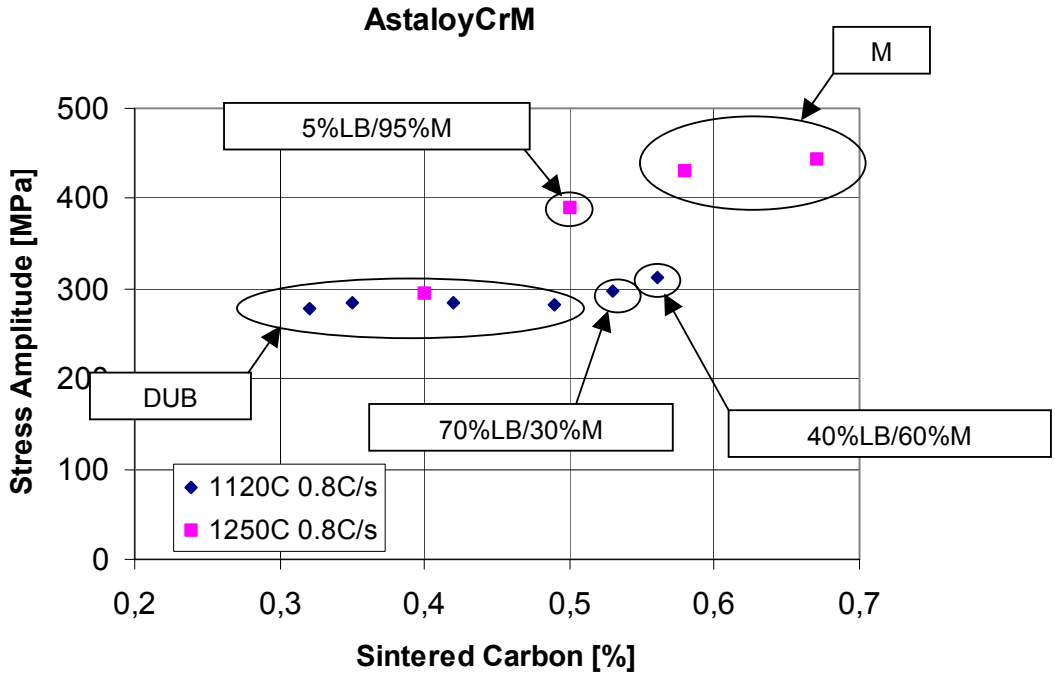


Figure 9. Fatigue performance of AstaloyCrM at different carbon contents. DUB: dense upper bainite, LB: lower bainite, M: martensite.

AstaloyCrL and AstaloyCrM show microstructure transition when the carbon level is increased. This transition is also depending on the cooling rate, i.e. the transition interval shifts to lower carbon values when the cooling rate is increased from 0.8°C/s (1.4°F/s) that is the cooling rate used here. Very high fatigue performance is obtained for AstaloyCrM independent of the microstructure. Choice of microstructure is therefore more a question of other demands like machinability, surface hardness, etc. Surface hardness HV10 of the four 1250°C(2280°F) sintered AstaloyCrM materials are shown in Table X. The results for AstaloyCrM with carbon level ≥0.58% show that high carbon and 0.8°C/s (1.4°F/s) cooling rate is a possible alternative to low carbon and high cooling rate for sinter hardening.

Table X. Surface hardness HV10 of 1250°C(2280°F) sintered AstaloyCrM

Powder	Sintering	Carbon As-Sint [%]	Density As-Sint [g/cm^3]	HV10
AstaloyCrM	1250°C, 30 min, 90/10 N_2/H_2 + 1120°C, 30min, 90/10 N_2/H_2 0.8°C/s	0.40	7.17	316
		0.50	7.15	454
		0.58	7.15	496
		0.69	7.16	496

About the same fatigue performance is obtained for diffusion-alloyed D.HP1 and AstaloyCrM with dense upper bainite or mixed lower bainite and martensite. However, two basically different strengthening mechanisms are active in the two materials. The martensitic network surrounding the base powder cores is important in PM steels with heterogeneous microstructures containing martensite in the sinter necks [1]. The suggested strengthening mechanism in D.HP1 is that transition to martensite induces compressive stresses in the martensite network where also the pores are contained. Corresponding tensile residual stresses must, of equilibrium reasons, be present in the non-martensitic regions, i.e. the upper

bainitic base powder cores. The strengthening mechanism in Chromium pre-alloyed AstaloyCrM and AstaloyCrL is solely obtained from strong microstructures.

The influence of shot peening on AstaloyCrL is very large. Comparative tests have been made on high temperature as-sintered AstaloyCrL+0.75%C and through hardened/250^{o}C(480^{o}F) vacuum tempered AstaloyCrL+0.5%C without and with shot peening. Both materials show extremely good response to shot peening. The fatigue limit is raised 79% for the as-sintered material after shot peening and 73% for the through hardened and tempered material. The influence of shot peening has been demonstrated for a notch sample opposed to plane bending. This is a geometry and load case where maximum influence of compressive residual stresses and shot peening can be expected. The influence on un-notched bars is most probably smaller.

CONCLUSIONS

- The control of microstructure results in high fatigue strength of materials based on pre-alloyed Chromium powders.
- The fatigue limit of materials based on AstaloyCrL is at 7.1 g/cm^3 230 – 250 MPa at carbon levels 0.6 – 0.8%C.
- The fatigue limit of materials based on AstaloyCrM is at density 7.05g/cm^3 280 – 445MPa at carbon levels 0.35 – 0.70%C.
- The fatigue limit of materials based on D.HP1 is at 7.1 g/cm^3 280 – 330 MPa at carbon levels 0.65 – 0.85%C.
- Shot peening of high temperature sintered AstaloyCrL with fine-pearlite microstructure increases the bending fatigue limit from 235 to 420 MPa.
- Shot peening of high temperature sintered/through hardened/250^{o}C(480^{o}F) tempered AstaloyCrL increases the bending fatigue limit from 285 to 490MPa.

REFERENCES

1. Anders Bergmark and Luigi Alzati, “Fatigue crack walk in Cu-Ni-Mo alloyed PM steel.” Accepted for publication in special issue of Fatigue and Fracture of Engineering Materials and Structures (FFEMS).