Influence of secondary operations on mechanical properties of low alloyed sintered steel

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Abstract
The fatigue performance of as sintered PM steel is depending on microstructure and density. Additional increase of the fatigue performance can be achieved by the secondary operations case hardening and/or shot peening. Shot peening is a well-established secondary operation on homogeneous steels with the purpose to introduce compressive residual stresses in the surface and to increase the yield stress by work hardening. Two additional features of shot peening are utilized in PM. Local plastic deformation of the surface layer increases the relative stiffness of the cross-section and also eliminates the detrimental effect of pores open to the surface. The present study is made on 1.5%Chromium/0.2%Molybdenum pre-alloyed PM steel notched samples in plane bending load mode. Increase of the plane bending fatigue performance by about 70% is found in cases where all mechanisms are combined.

Introduction
The expansion of the potential market for PM steel is closely related to improved mechanical performance of the materials. Increased fatigue performance is a key issue and this can be achieved by introduction of strong microstructures and smaller pores [1,3].
Case hardening and shot peening are secondary operations, which further improve the fatigue performance. [1,2,3] The aim of these methods is to introduce gradient material properties and residual stresses on the macroscopic level [2,3]. Case hardening introduces martensite in the surface. Compressive residual stresses are introduced in the martensitic parts because of the relatively increased volume of martensite compared to other possible microstructures at room temperature. High carbon martensite has lower density than low carbon martensite and a residual stress distribution can therefore be present in a fully martensitic material provided there is a carbon gradient. Shot peening also gives residual compressive stresses in the surface.
Fatigue performance of PM steels is depending mainly on the maximum tensile stress obtained from the combination of the residual stress distribution and the stress field from the outer load. Compressive residual stresses in the surface must of equilibrium reasons be balanced by tensile residual stresses in other parts of the component. A successful utilization of introduction of compressive residual stresses means that compressive residual stresses appear in areas with high tensile stresses from the outer load and tensile residual stresses in areas with low stresses. Largest influence of secondary operations can be expected for components with sharp stress gradient. The experiments in this investigation were made on 3 mm radius notch bar in plane bending.

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Experimental

Test specimen

The test bar design is shown in Fig.1. Width *w* and height *h* are both equal to 7.0 mm. The minimum cross-section has chamfered edges along the 3 mm notch [1,4].

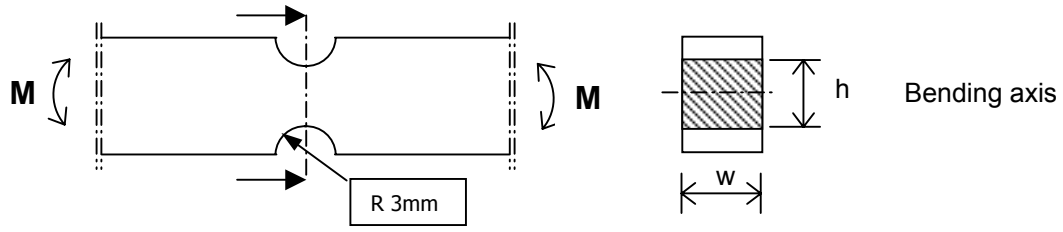


Fig. 1. Test bar geometry for all fatigue tests.

The 3 mm radius notched test bar has theoretical stress concentration K_t=1.38. The stress distribution obtained from FEM calculation is shown in Fig. 2.

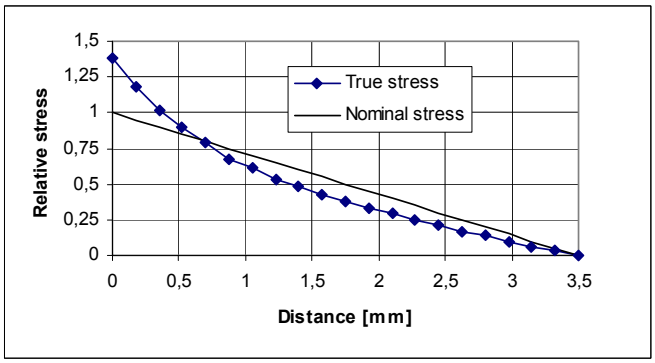


Fig. 2. Relative stress distribution in 3 mm radius 7x7 mm^2 test bar.

Material

Test bars of the pre-alloyed powder Astaloy CrL (1.5%Cr, 0.2%Mo, rest Fe) with addition of graphite (Kropfmühl C-UF4) and 0.6% lubricant were warm-compacted at 800MPa. Sintered density was around 7,3 g/cm^3.

Sintering

Sintering was carried out at 1280^{o}C in either N_2 or H_2. The N_2-sintering was made in a continuous high temperature furnace of the type C/C (Carbon fiber reinforced Carbon Composite) [5,6] made by the company KYK in Japan. The H_2-sintering was made in a laboratory batch furnace. Cooling rate in both cases was around 0.7^{o}C/s.
Depending on the secondary operations different combined carbon contents were targeted after sintering. Materials to be case hardened had a carbon content after sintering of 0,2% whereas those for through hardening had 0,5% and those for testing in the as-sintered condition had 0,7% combined carbon.

Case hardening

Case hardening was performed by two different methods. One by gas carburization followed by oil quenching and the other by low pressure carburization followed by gas quenching (N_2,

10bar). Case depth was 0.45mm for gas carburized materials and 0.6mm for low pressure carburized materials. All case hardened materials were tempered at 200°C for one hour in air.

Through hardening
Samples with 0.5% Combined carbon were through hardened by gas quenching. Tempering was made at 250°C for one hour. The relatively high tempering temperature was chosen in order to increase ductility and the possibility to get plastic deformation on the surface from shot peening.

Shot peening
The shot peening parameters are shown in Table I.

Type	Air nozzle
Shot	Steel balls
Air pressure	0.3MPa
Peening time	15sec
Distance to work	150mm
AlmenA intensity	0.32mm
Work revolutions	12-15rpm
Coverage	200-300%

Table I. Shot peening parameters.

Fatigue tests
Tests were performed by plane bending. All tests were made in displacement control with load ratio R=-1, run-out limit 2 million load cycles and test frequency 27-30 Hz. Fatigue limits were evaluated with the staircase method according to MPIF standard 56 or estimated directly from the S-N diagrams. The maximum load capacity of the fatigue test system was 490MPa nominal stress for the specimen geometry used in this investigation. Regular staircase tests could not be performed for the high capacity materials as run-outs were obtained at the highest allowed stress level.

Micrographs
Microstructures were evaluated by Light Optical Microscopy (LOM). SEM fractography was used to trace crack initiation in samples after secondary operations.

Results
Astaloy CrL+0.70%C –no heat treatment
The microstructure is characterized by fine pearlite. As sintered material has a nominal stress fatigue limit of about 235MPa (estimated from the S-N curve) corresponding to maximum theoretical stress of 1.38 x 235 = 324MPa. Shot peened samples show a fatigue limit of 415MPa nominal stress corresponding to 572MPa maximum theoretical bending stress. Shot peening increases the fatigue performance by 77%.

The thickness of the almost fully densified surface layer obtained from shot peening is about 100 μm. Fractography of two samples shows crack initiation 0.5mm below the surface (fish eye fracture) at a large pore and on the surface.

Astaloy CrL+0.50%C –through hardened
All samples in this series were through hardened by gas quenching and tempered at 250°C. The microstructure is characterized by tempered martensite. Slight densification of the surface layer from shot peening is found down to 50 μm below the surface. The nominal fatigue limit

was about 285MPa (estimated from the S-N curve) corresponding to a maximum theoretical stress of 389MPa. Shot peening increased the fatigue limit to > 490MPa nominal stress (the test system did not allow higher values) corresponding to > 670MPa maximum theoretical bending stress. Fractography of one sample showed crack initiation 300 μm below the surface. In this case, shot peening has increased the plane bending fatigue performance from about 285MPa to >490MPa, corresponding to >72%.

Case hardened Astaloy CrL
Results from the fatigue tests are shown in Table II.

Sintering	Carburizing	Quenching	Shot peening	Surface hardness HV01	Case depth [mm]	Fatigue limit [MPa]	
						Nominal	Maximum
N2	Gas	Oil	NO	800	0.45	407	562
			YES	NA		415	572
N2	Low pressure	Gas	NO	750	0.60	363	501
			YES	NA		>470*	>621
H2			NO	750		386	533
			YES	NA		>470*	>621

Table II. Case depths and results from fatigue tests. Maximum stresses are 1.38 times nominal stresses. NA: not available. *: Only a few samples have been tested. No broken samples have been obtained at test level 470MPa. The fatigue limit is estimated to >470MPa.

Gas carburized and oil quenched materials were fully martensitic with lath martensite in the core and martensite with small amounts of retained austenite in the surface. Oxidation in grain boundaries was found down to 200 μm from the surface.
Low pressure carburized and gas quenched materials had bainitic cores and martensite + small amounts of carbides and retained austenite in the surface. The distribution of retained austenite was, however, uneven. More retained austenite was found in the middle of the die surface (around the compaction neutral layer).

Discussion
The influence of shot peening on the Chromium/Molybdenum pre-alloyed Astaloy CrL is shown to be very strong. In addition to the introduction of compressive residual stresses from shot peening and work hardening of the surface layer two more mechanisms that cannot be utilized in homogeneous materials are present in PM steels, contributing to the strong response on fatigue performance. The two mechanisms unique for PM are the relative increase of the surface stiffness that due to the densification reduces the strain in the entire cross-section at a given load level and the elimination of the detrimental effect of pores open to the surface.
The mechanisms can be quantified for the test bars used in this investigation. The first mechanism moves the maximum total (from residual stress distribution and outer load) tensile stress down [3]. Assume that maximum tensile stress is obtained 0.3 mm below the surface. An inspection of Fig. 2 indicates that the relative stress is reduced from 1.38 to about 1.0.
The second mechanism, i.e. the difference between influence of pores open to the surface and embedded pores is evaluated from fracture mechanics. The basic assumption is that pores behave like cracks with crack-size equal to the projected area of the pore and that the fatigue limit is determined from the (threshold) stress intensity factor. Compare a half-circle (projected) pore open to the surface with radius *ro* and an embedded circular crack with radius *re*. Equal crack areas gives $ro^2 = 2re^2$. The stress intensity factor from small cracks at or close to the surface is $K_I=0.67\sigma\sqrt{\pi a}$, [7] where σ is remote stress and *a* is the crack radius. Equal stress intensity factor for the embedded and surface cracks gives $\sigma e=2\text{^}0.25\cdot\sigma o \approx 1.19\ \sigma o$.

Increased cross-section stiffness from a 100 μm fully densified surface layer (typical for a non martensitic surface) is about 10% (calculations are not shown here). The possible theoretical cumulative influence from the mechanisms is in this case 1.38 x 1.19 x 1.1 ≈ 1.81 for non-martensitic material and 1.38x1.19 ≈ 1.64. The results obtained here were 1.77 for as sintered ductile material and 1.72 for through hardened/annealed material. The model is valid for materials with homogeneous microstructures and no compressive residual stresses present before the shot peening operation.
Gas carburized/oil quenched/200^{o}C tempered material shows a fatigue limit of 407MPa. This is close to fatigue performance of case hardened materials based on Astaloy Mo (1,5%Mo rest Fe) +0.2%graphite with a case depth of 0.9mm reported by Gunnar Åkerström [8]. Additional shot peening of gas-carburized case hardened Astaloy CrL increases the fatigue limit slightly to 417MPa.
Low-pressure carburized test bars show lower fatigue performance after case hardening only and higher after shot peening compared to the gas-carburized bars. The differences in microstructure and case depth are shown in Table III.

		Gas carburized	**Low pressure carburized**
Surface	Oxidation	Down to 200 μm	NO
	Retained austenite	Small amounts	YES
	Carbides	NO	Small amounts
	Case depth	0.45mm	0.6mm
Core microstructure		Lath martensite	Bainite

Table III. Comparison of microstructure features of gas carburized and low pressure carburized Astaloy CrL

Possible reasons for the lower fatigue performance after case hardening are different case depths and presence of carbides and retained austenite. It is noticed that presence of oxides close to the surface in gas-carburized materials seems not to be detrimental for the fatigue performance. A few of the low-pressure carburized/shot peened samples have been fatigue tested. The results clearly indicate that the influence of shot peening is strong also for this material. The fatigue limit is above 470MPa nominal stress and brings the material to about the same level as the through hardened/tempered/shot peened material.
The fatigue performances of the microstructures that can be processed in Astaloy CrL are high [1] and secondary operations bring the material to even higher performance. One important result is that as sintered Astaloy CrL with a microstructure characterized by fine pearlite can be improved by shot peening to a nominal stress fatigue limit of 415MPa corresponding to a maximum theoretical stress of 573MPa. This is a very high value and the material is a candidate for high fatigue loaded components where wear resistance and a very hard surface obtained from martensite are not necessary.
The highest performance, exceeding 490MPa nominal stress and 670MPa theoretical maximum stress fatigue limit, is found for through hardened/tempered/shot-peened material. The secondary operations have not been optimized in this investigation. The choice of case depth and shot peening parameters were based on mechanical analysis of the stress distribution. Choice of shot peening parameters was based on experience from shot peening of homogeneous steels [9].
The combination of high performance microstructures obtained in sintered Chromium/ Molybdenum pre-alloyed PM steel, a secondary hardening operation and shot peening increase the fatigue performance considerably. This brings PM steel close to homogeneous high performance steel and widens the potential for PM steel applications where high fatigue performance is the key factor.

Conclusions

The following results were achieved with a material based on Astaloy CrL sintered at 1280°C and a core density of 7,3 g/cm^3:

- Shot peening with fine-pearlite microstructure increases the nominal bending fatigue limit from 235 to 420MPa.
- Shot peening of through hardened/250^{o}C tempered material increases the bending fatigue limit from 285 to >490MPa.
- Gas carburized/oil quenched case hardened and tempered material with lath martensite in the core and martensite with small amounts of retained austenite in the surface has a fatigue limit of 407MPa. Only a small positive influence of shot peening was found.
- Low-pressure carburizing/gas quenched and tempered material with bainite core and martensite with retained austenite in the maximum loaded surface layer has a fatigue limit of 363 – 389MPa. Shot peening brings the material to a fatigue limit exceeding 470MPa.

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