# Fatigue performance of PM steels in as-sintered state.

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## Abstract

An overview of the plane bending fatigue performance of PM steels in as-sintered state is presented. Pre-alloyed Molybdenum and Chromium-Molybdenum materials, diffusion-bonded Ni-Cu-Mo and Fe-2%Cu pre-mixes are reviewed. The effect of varying density level and Carbon content upon fatigue performance is analyzed separately. Diffusion-bonded materials exhibit heterogeneous microstructures with high performance metallographic phases distributed along the sinter-necks and around the pores. Optimized carbon content in diffusion-alloyed materials is characterized by a well-distributed martensitic microstructure. Furthermore, fatigue crack initiation is found in the low yield stress phase in the base powder core. PM steels based on pre-alloyed powders exhibit one phase or random distribution of two or more phases with no systematic order in relation to the pores, hence fatigue crack initiation is related to sinter-neck failure. Influence of density is more pronounced for pre-alloyed materials than compared to diffusion-bonded materials.

## Introduction

Microstructure and Density are the most relevant parameters influencing PM steels fatigue performance. The comprehension of materials' microstructure allows the interpretation of fatigue properties [1, 2, 3].
The role of microstructure is analyzed in this paper by the comparison of different alloying systems and different alloying techniques: pre-alloyed, premixed and diffusion-bonded powders at density 7,1g/cm$^3$ are reviewed. Two Carbon levels have been considered for each material.
The role of density is shown for materials based on one pre-alloyed and two diffusion bonded powders. Various compaction techniques have been used.
All the materials have been processed by the same sintering route: 1120°C/30', 0,8°C/s the cooling rate. One example of sinter-hardened material with 2,5°C/s cooling rate has also been included.

## Materials

Table 1 lists all the investigated materials. Powders have been mixed with 0.8% Amid-wax, cold compacted at 7.1-7.15g/cm$^3$, sintered in belt furnace at 1120°C in 90/10 $N_2/H_2$

atmosphere for 30’, with 0.8°C/sec cooling rate. One diffusion-bonded material has also been tested in sinter-hardened conditions with 2,5°C/s cooling rate.

Table 1. Chemical composition and alloying technique for all the investigated iron-based powders.

| Group | Material | Pre-alloyed elements | Diffusion Bonded elements | Premixed elements |
|---|---|---|---|---|
| Pre-alloyed | Astaloy85Mo | 0,85%Mo | - | - |
| | AstaloyMo | 1,5%Mo | - | - |
| | AstaloyCrL | 1,5%Cr, 0,2%Mo | - | - |
| Premixed | ASC100.29 | - | - | 2%Cu(-100mesh) |
| | Astaloy85Mo | 0,85%Mo | - | 2%Cu(-325mesh) |
| | Astaloy85Mo | 0,85%Mo | - | 2%Ni(Inco123) |
| Diffusion-bonded | DistaloyDC | 1,5%Mo | 2%Ni | - |
| | DistaloyAE | - | 4%Ni, 1,5%Cu, 0,5%Mo | - |
| | DistaloyHP | 1,5%Mo | 4%Ni, 2%Cu | - |
| | DistaloyDH | 1,5%Mo | 2%Cu | - |

## Experimental procedures

Four point bending fatigue tests have been performed for as-sintered, un-notched ISO 3928 test bars with modified cross section [1]. The following test parameters have been used: load ratio R =-1 (i.e. mean stress equal to zero); frequency: 25-29 Hz; broken specimens stopped for 1.5% increased compliance; run out at 2 millions cycles; fatigue endurance [$\sigma_{A,50\%}$] and standard deviation were determined by the staircase method, according to MPIF Standard No.56, 2001. The staircase step has in all cases been 10MPa.
Ordinary Light Optical Microscopy (LOM) observations have been made on polished and etched samples in order to analyze materials’ microstructures.
Scanning electron microscopy (SEM) fractography observations have been made on specimens tested at stress levels less than 5% higher than the $\sigma_{A,50\%}$ value of the respective material.

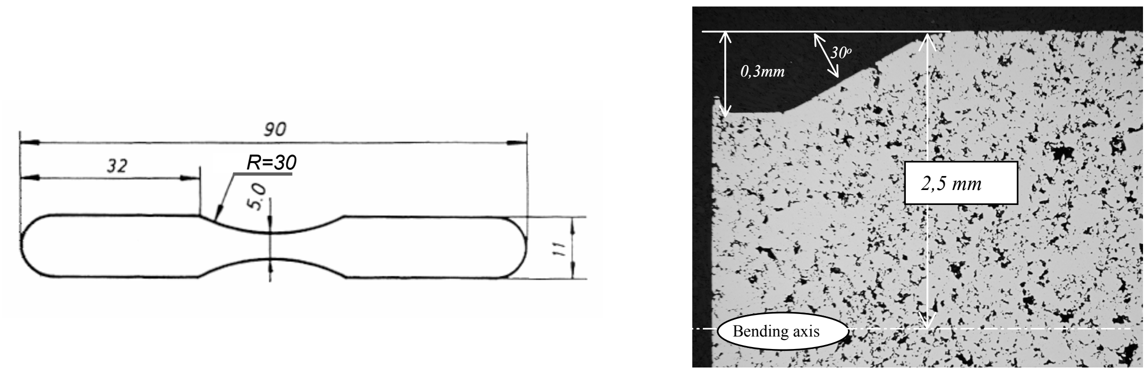


Figure 1. Modified ISO 3928 test specimen with chamfered edges.

## Fatigue tests results

### Density close to 7,1 g/cm³

All the results obtained for materials cold compacted at density close to 7,1g/cm$^3$ are summarized in Table 2.

Table 2. Bending fatigue endurance evaluation at 2 million cycles, according to MPIF Standard No.56 (2001) for as-sintered materials at 7,1g/cm$^3$ density. Sintering performed in belt furnace at 1120°C in 90/10 $N_2/H_2$ atmosphere for 30', 0.8°C/sec cooling rate, except where noted.

| Group | Material | Carbon [%] | $\sigma_{50\%}$ [MPa] | Std Dev [MPa] |
|---|---|---|---|---|
| Pre-alloyed | Astaloy 85Mo | 0,48 | 180 | 11 |
| | Astaloy 85Mo | 0,76 | 196 | 8 |
| | Astaloy Mo | 0,42 | 201 | <5 |
| | Astaloy Mo | 0,82 | 210 | <5 |
| | Astaloy CrL | 0,55 | 236 | 8.5 |
| | Astaloy CrL | 0,74 | 252 | <5 |
| Premixed | ASC100.29 +2%Cu (-100) | 0,49 | 190 | <5 |
| | ASC100.29 +2%Cu (-100) | 0,77 | 220 | <5 |
| | Astaloy 85Mo +2%Ni | 0,5 | 201 | 16 |
| | Astaloy 85Mo +2%Ni | 0,78 | 210 | <5 |
| | Astaloy 85Mo +2%Cu(-325) | 0,49 | 211 | 15 |
| | Astaloy 85Mo +2%Cu(-325) | 0,78 | 233 | <5 |
| Diffusion-Bonded | Distaloy AE | 0,46 | 224 | 7 |
| | Distaloy AE | 0,81 | 274 | 8.6 |
| | Distaloy DC | 0,47 | 230 | <5 |
| | Distaloy DC | 0,83 | 239 | <5 |
| | Distaloy HP | 0,48 | 251 | 13 |
| | Distaloy HP | 0,85 | 326 | <5 |
| | Distaloy DH | 0,49 | 209 | 15 |
| | Distaloy DH | 0,76 | 236 | 22 |
| | Distaloy DH (2,5°C/s) | 0,47 | 236 | 13 |
| | Distaloy DH (2,5°C/s) | 0,73 | 271 | 17 |

### Densities higher than 7,1 g/cm³

Bending fatigue tests have been performed for Diffusion-bonded Distaloy AE and Distaloy DC and pre-alloyed Astaloy CrL at different density levels. One Carbon level has been set for each material, in order to keep microstructure as constant parameter. Cold compaction, warm compaction, double pressing double sintering and powder forging have been performed to obtain the four density levels shown in Figure 2.

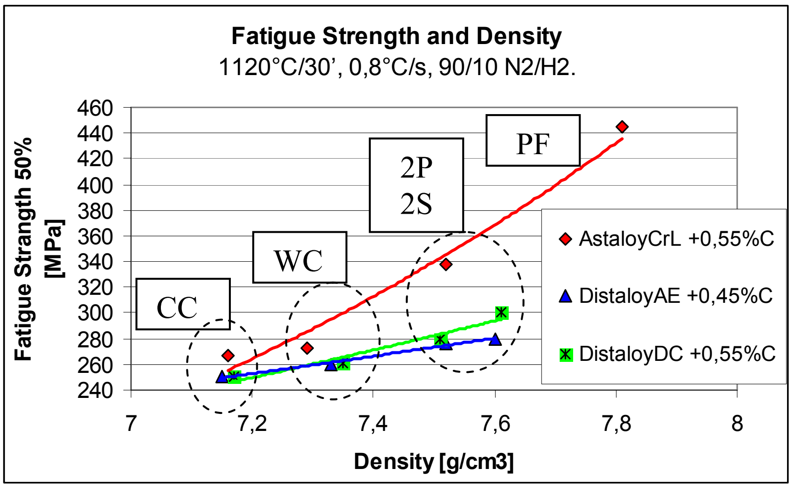


Figure 2. Bending fatigue endurance evaluation at 2 million cycles, according to MPIF Standard No.56 (2001) for as-sintered materials at different densities. Sintering performed in belt furnace at 1120°C in 90/10 $N_2/H_2$ atmosphere for 30', 0.8°C/sec cooling rate. Four levels of density have been achieved by Cold Compaction (CC), Warm Compaction (WC), Double pressing double sintering (2P2S), Powder Forging (PF).

## Metallography

### Pre-alloyed materials

Astaloy85Mo shows upper bainitic structure at 0,48%C; at 0,76%C spots of very dense upper bainite can also be detected. AstaloyMo shows upper bainitic structure at 0,42%C, while at 0,82%C also spots of lower bainite can be detected [3].
AstaloyCrL shows upper bainitic and pearlitic structure at 0,55% and 0,74%C respectively [2]. Upper bainite is denser in AstaloyCrL than in AstaloyMo and even more than in Astaloy85Mo, at similar Carbon levels; i.e. the ferritic areas among cementite lamellas are the smallest for Astaloy CrL.

### Premixed materials

Standard Fe+2%Cu mixes show ferritic-pearlitic structure. Cu-rich areas surround the base powder particles.
Astaloy85Mo +2%Ni (Inco123) +0,5%C shows upper bainitc base powder particles and austenitic Ni-rich islands partially surrounded by spots of martensite. At 0,78%C, the base powder particles show denser upper bainite; still the austenitic islands do not show a continuous martensitic shell.
Astaloy85Mo +2%Cu(-325) shows fully upper bainitic structure at 0,49%C; spots of denser upper bainite [~30x30$\mu m^2$] can be detected in correspondence of the Cu-rich areas. At 0,78%C the base powder particles show denser upper bainite and spots of lower bainite/martensite [~50x50µm] are present in the Cu-rich areas.

Diffusion-bonded materials

DistaloyAE shows ferritic/pearlitic and fully pearlitic base powder particles at 0,46% and 0,81%C respectively. A Cu-Ni-rich network surrounding the base powder particles is consisting of lath martensite/lower bainite with austenitic islands and plate martensite with austenitic islands at 0,46% and 0,81%C respectively.
Both Distaloy DC and Distaloy HP show upper bainitic base powder particles at Carbon levels close to 0,5% and 0,8%. Ni-rich austenitic islands in Distaloy DC show thicker martensitic shells for higher Carbon level. Cu-Ni-rich network in Distaloy HP shows the same characteristics that are seen for Distaloy AE [3] –see Figure 3.
As-sintered Distaloy DH shows fully upper bainitic structure at 0,49%C, while at 0,76%C lower bainitic/martensitic spots can be detected in correspondence of some sinter-necks. It must be pointed out that no continuous martensitic network is established.
Sinter-hardened Distaloy DH shows at 0,47%C upper bainitic base powder particles and lower bainitic/martensitic Cu-rich network; at 0,73%, the base powder particles show mainly martensitic structure, with bainitic core. The Cu-rich network is martensitic –see Figure 3.

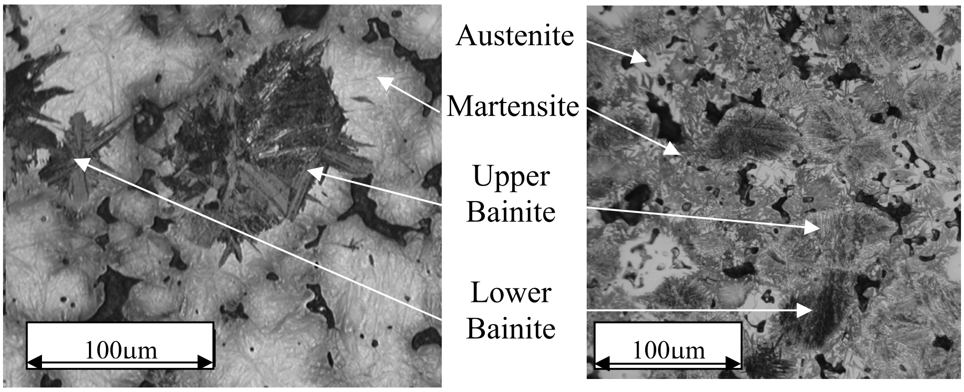


Figure 3. To the left: Microstructure of Distaloy DH (2,5°C/s) +0,73%C. To the right: microstructure of Distaloy HP +0,85%C. At density close to 7,1g/cm$^3$, these microstructures allowed $\sigma_{50\%}$ equal to 271MPa (326MPa) and Standard deviations equal to 17MPa (<5MPa) for Distaloy DH (Distaloy HP).

## Fractography

SEM fractography reveals different behavior of diffusion-bonded materials, compared to pre-alloyed materials. The latter show crack early propagation mainly developed by inter-particles modality, i.e. through the sinter-necks –see Figure 4. In crack initial propagation area Distaloy materials show mainly trans-particles fracture, i.e. through the base powder particles –see Figure 5.

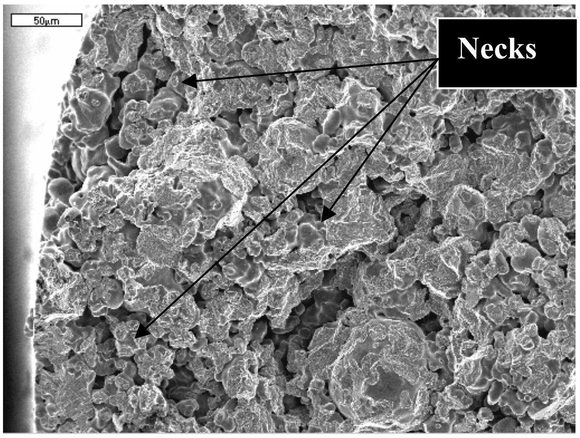


Figure 4. Crack initiation and early propagation area for Astaloy Mo +0.84%C. Mainly inter-particles fracture, i.e. through the sinter-necks.

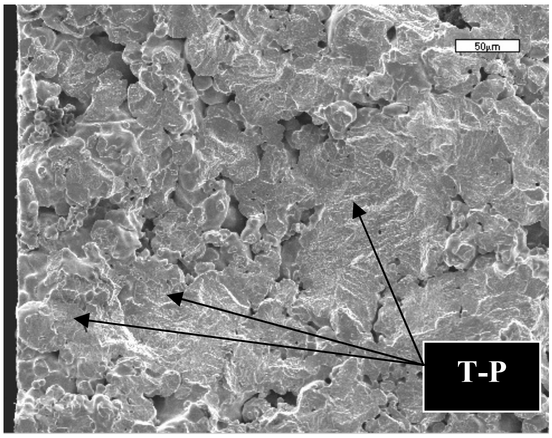


Figure 5. Crack initiation and early propagation area for Distaloy HP +0.65%C. Mainly trans-particles fracture ("T-P"), i.e. through the base powder particles.

# Discussion

## Fatigue Tests results

*Pre-alloyed materials*
Different levels of density of upper bainite (i.e. amount of ferrite among the cementite lamellas) give reason for the different performance of AstaloyCrL, AstaloyMo and Astaloy85Mo. Tests results for the high Carbon level series show better performance of AstaloyCrL pearlitic structure, compared to upper bainitic Astaloy85Mo and AstaloyMo.

*Premixed materials*
2%Ni and 2%Cu additions increased the performance of Astaloy85Mo by 10% and 15% respectively.
Due to the better performance of upper bainitic compared to ferritic/pearlitic base powder particles, Astaloy85Mo +2%Cu(-325) shows 7% to 10% higher fatigue strength than ASC100.29 +2%Cu(-100). Smaller Copper particles size might also have played a beneficial role.

*Diffusion-bonded materials*
DistaloyAE shows microstructural modification level both in the base powder particles and in the Cu-Ni-rich network with increasing Carbon level from 0,46% to 0,81%: the reduction of the ferritic portion in the ferritic/pearlitic base powder particles and the transformation to fully plate martensite of the Cu-Ni-network has beneficial effect on the fatigue strength.
Increased Carbon level from 0,47 to 0,83% has limited effect on the microstructure of Distaloy DC; consequently, the fatigue strength shows only slight difference (~3,5%).

The beneficial role of optimized network microstructure is highlighted by Distaloy HP: from 0,48% to 0,85%C no significant difference is observed in the bainitic base powder particles; therefore, the 30% increase in fatigue strength is to be considered due to the formation of fully plate-martensitic Cu-Ni-network.
As-sintered DistaloyDH shows similar microstructure to Astaloy85Mo +2%Cu(-325), at comparable Carbon levels. The fatigue performance is therefore similar. It has to be noticed that fatigue strength values are slightly lower than what obtained for DistaloyDC: the latter presents formation of martensite in small areas in correspondence of the Ni-rich sinter-necks.
Sinter-hardened Distaloy DH +0,47%C shows mostly-martensitic/lower bainitic Cu-rich network. The amount of lower bainite is so high that the same performance of as-sintered Distaloy DH +0,76%C is achieved.
Sinter-hardened Distaloy DH +0,73% shows 15% higher fatigue strength, compared to what found at 0,47%C. This result is remarkably lower than what shown by Distaloy HP. The reason has to be found in the microstructural modification due to increased Carbon level: at 0,73%C martensite is present not only in the network, but also in large portions of base powder particles. This structure is less favorable for fatigue compared to the thinner, fully plate-martensitic and thinner network shown by Distaloy HP +0,85%C –see Figure 3.
In Distaloy materials the effect of 2%Ni is higher than the one of 2%Cu (compare Dsitaloy DC to as-sintered Distaloy DH); the opposite result was found in premixes of Astaloy85Mo. The reason is the pronounced martensitic transformation occurring in Ni-rich areas in the diffusion-bonded Distaloy DC, due to better diffusion of Nickel, combined with higher Molybdenum level in the base powder.

Scatter in fatigue strength results

Pearlitic and bainitic pre-alloyed materials (Astaloy CrL, Astaloy85Mo and AstaloyMo) show scatter in fatigue strength (standard deviation) lower than 5%. This result is comparable with what found for bainitic dense steels [5]. Slightly higher value has been found for Astaloy85Mo +0,48%C, probably due to the fact that large areas of ferrite among cementite lamellas acting as weak phase.

ASC100.29 +2%Cu(-100mesh) shows very low standard deviation (<3%): the heterogeneous ferritic/pearlitic structure of base powder particles is surrounded by a reinforced Cu-rich shell. The low scatter indicates that these phases have similar yield strength.
In premixes of Astaloy85Mo with Copper or Nickel very low scatter is shown at Carbon levels close to 0,8%. Standard deviations of respectively 7% and 8% are generated at Carbon level close to 0,5%, probably due to the presence of inhomogeneous distribution of the metallographic phases (austenitic/martensitic islands and dense upper bainitic areas in mixes with 2%Ni and 2%Cu respectively).

In Distaloy materials scatter is generated when a continuous high yield phase network is not formed (as-sintered Distaloy DH) or is not fully consisting of martensite (Distaloy HP +0,48%C, sinter-hardened Distaloy DH +0,47%C). In these cases the scatter is between 5 to 10%.

For sinter-hardened Distaloy DH +0,73%C upper bainitic islands act as weak points in the extended portion of material that is transformed into martensite and are responsible for the ~6% standard deviation.

Fatigue fracture behavior and response to high density

Homogeneous PM steels are characterized by equal phase composition in the powder particles and in the sinter-necks. Due to a phenomenon of stress concentration, crack initiation is related to sinter-necks failure, i.e. the material is sensitive to porosity level. Therefore, the increase of density is beneficial for homogeneous PM steels (Astaloys).

Heterogeneous PM steels with sinter necks effectively reinforced by the presence of a continuous martensitic network (DistaloyAE, DistaloyDC, DistaloyHP, Astaloy85Mo+2%Cu, sinter-hardened Distaloy DH) present crack initiation mostly related to base powder particles failure, whose microstructure (ferrite/pearlite or bainite) has lower yield threshold compared to martensite [4, 6]. With regards to sensitivity to porosity, the sinter-necks are optimized by the presence of martensite. Increase of density will therefore have less significant effect compared to homogeneous PM steels, with regards to fatigue endurance.

## Conclusions

- Microstructure analysis gives reason for PM steels fatigue strength values and the relative scatter. Fractography is a useful method to reveal materials' sensitivity to porosity.

- Sinter necks are effectively reinforced once that a continuous high yield microstructure network is formed among the base powder particles.

- Increasing density is beneficial on the fatigue performance of homogeneous PM steels (Astaloys) and not effective on materials characterized by network-like microstructures (Distaloys).

References


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