MICROSTRUCTURAL REINFORCEMENT OBTAINED BY DIFFUSION BONDING.

Luigi Alzati, Anders Bergmark, Jessica Andersson,

Höganäs AB SE 26383 Höganäs, Sweden

ABSTRACT

Diffusion bonding is an effective method to obtain high fatigue performance in PM steels. The main characteristic of diffusion-bonded materials consists of a well-organized microstructural heterogeneity. This work gives reason of the mechanism underlying this fact by means of microstructural interpretation of dynamic performance.
Four point bending fatigue tests have been performed on D.AE and D.HP powder metal steels at density close to 7.1 g/cm^3, sintered at 1120°C (2050°F); cooling rate was 0.8°C/s (1.4°F/s). Notch sensitivity has been tested with 0.25, 0.9 and 3 mm notch radius.
Particular focus has been made on the interaction of the different phases. The effect of the Cu-rich network surrounding the base powder particles is considered to be the main factor responsible for the high fatigue performance of diffusion-bonded materials.

INTRODUCTION

The main goal of this work is to provide systematic information to designers with regards to the notch sensitivity of diffusion bonded PM steels.
Previous works on the same materials here investigated have been performed on test specimens where the notch had been obtained by machining [1, 2, 3]. In this investigation bending fatigue tests have been performed on un-notched and 3, 0.9 and 0.25mm notched bars obtained by specifically designed compacting tools, in order to avoid influence of machining on the fatigue performance (results for 3mm notched bars are taken from reference [4]). Indication of the fatigue strength ($\sigma_{50\%}$) is given by means of staircase method calculation and verification of the plateau in the fatigue curve (σ/N) at stress levels close to $\sigma_{50\%}$.
On a general point of view, fatigue performance of a PM steel component depends on three main factors: the applied fatigue stress, the PM material strength and the presence of stress concentration phenomena. The latter is mostly related to design and surface conditions. PM steel fatigue strength is the outcome of a

combination of microstructure, density level and presence of internal residual stresses –i.e. not depending on design of the component.
Two of the factors affecting fatigue performance are discussed here: internal residual stresses, due to microstructural heterogeneity, and stress concentration due to specimens' design (notches) and surface treatment.
Bending fatigue tests have been performed on three series of materials in as sintered conditions. Microstructure has been analyzed by Light Optical Microscopy (LOM).
This work is focused on the bending fatigue behavior of two heterogeneous PM steels. Material D.AE is obtained by diffusion bonding of 4%Nickel, 0.5%Molybdenum and 1.5%Copper to pure iron base particles. Material D.HP is obtained by diffusion bonding of 4%Nickel and 2%Copper to 1.5%Mo pre-alloyed base particles. Microstructure has been set as fixed parameter: one only Graphite level (0.75%) and one only processing route have been adopted: the materials have been compacted to density close to 7.1g/cm^3 sintered at 1120°C (2050°F) in 90%N_2/10%H_2 atmosphere. Cooling rate was 0.8°C/s (1.4°F/s). Heterogeneous microstructure is obtained in the materials studied here exploiting the low diffusion rates into steel presented by Copper and Nickel at this sintering temperature. In particular, Copper melts at 1084°C (1983°F) and the transient liquid phase surrounds the base powder particles during sintering. Nickel diffuses into the Cu-melt and (more slowly) into the base powder particles. On a fatigue performance point of view, the resulting microstructure after cooling consists of a well-designed heterogeneity: a network of martensite is formed in the regions that are Cu/Mo-rich; in this way the sinter necks result reinforced [5, 6]; Nickel-rich areas result austenitic, being surrounded by a martensitic shell they can play beneficial role to arrest fatigue cracks [7]; base powder particles of material D.AE and D.HP are pearlitic and upper bainitic respectively.

EXPERIMENTAL PROCEDURES

Materials and microstructures

Table 1 shows the alloying systems corresponding to the PM steels investigated in this study. Table 2 shows the compacting pressure and the macro-hardness values. All materials have been sintered at 1120°C(2050°F) in 90%N_2/10%H_2.

Table 1. Composition of the investigated mixes.

Material	Diffusion Bonded elements			Pre-alloyed Elements	Mixed	
	Ni [%]	Cu [%]	Mo [%]	Mo [%]	C-UF4 [%]	Wax [%]
D.AE	4%	1.5%	0.5%	-	0.75	0.8
D.HP	4%	2%	-	1.5%	0.75	0.8

Table 2. Compacting pressure, sintered density and macro-hardness.

Material	Compacting Pressure [MPa]	Sintered density [g/cm^3]	HV10
D.AE	600	7.1	252
D.HP	640	7.1	314

Fatigue Bars

Four different fatigue bar designs have been adopted: ISO 3928 un-notched bars (named here FS) and 3, 0.9 and 0.25mm notched bars (named here N3, N0.9 and N0.25). N3 bar geometry is taken from reference [4]. Figure 1 and Figure 2 show in detail the sizes of the fatigue bars.

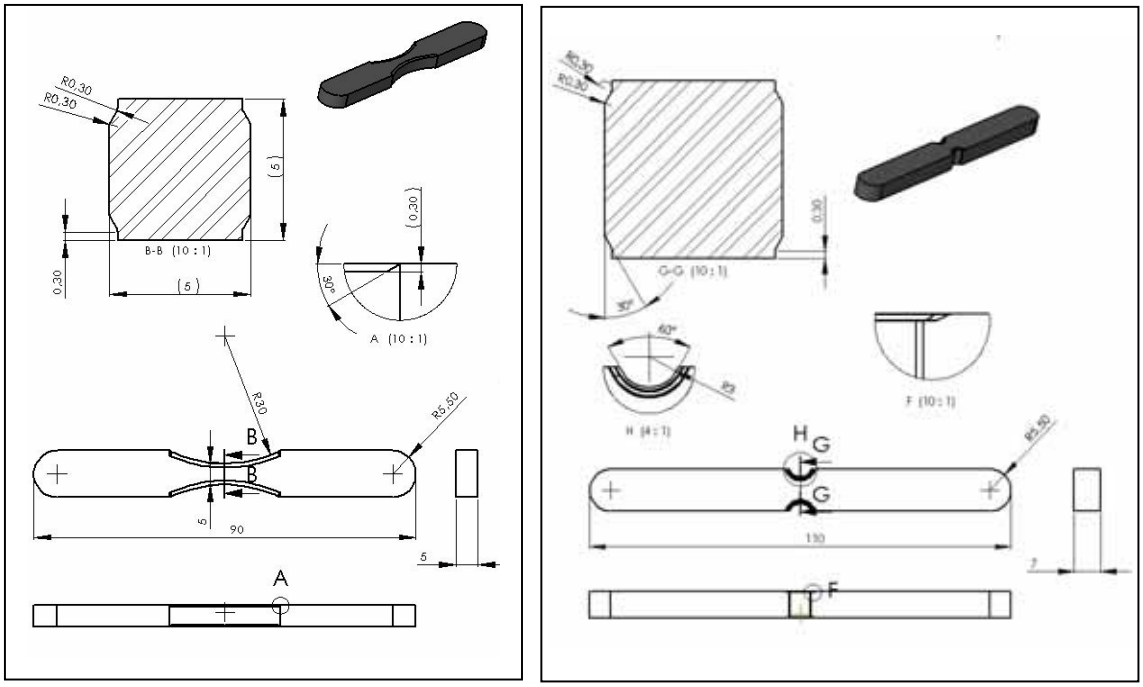


Figure 1. To the left: un-notched fatigue bar –'FS'. To the right: 3mm notched bar, 'N3', from [4].

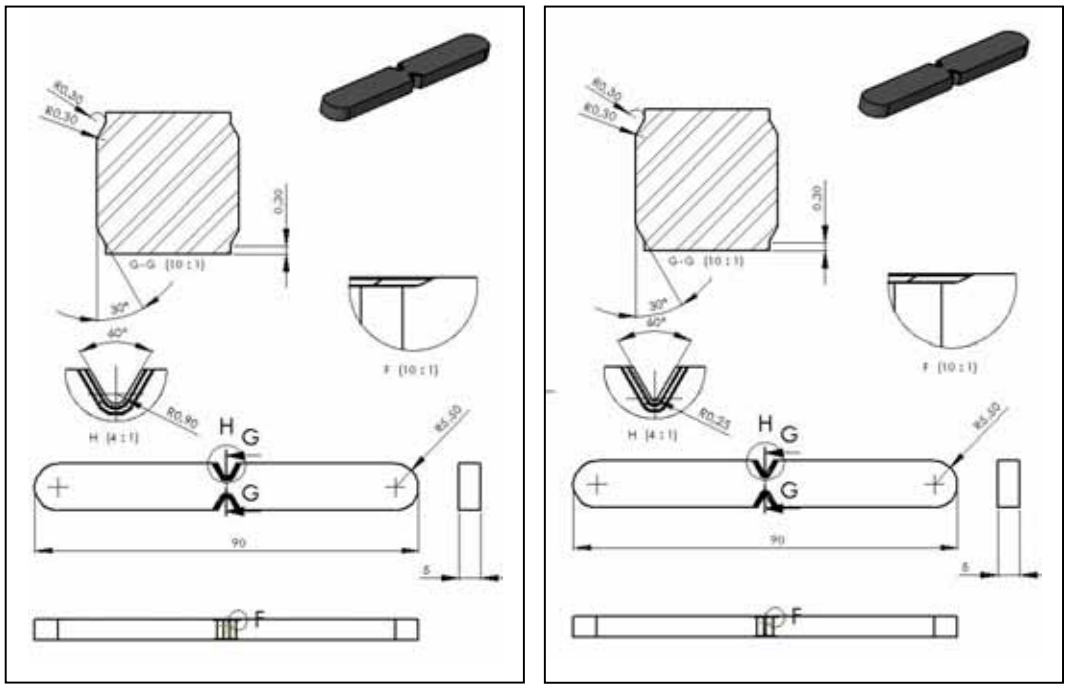


Figure 2. To the left: 9mm notched bar, 'N0.9'. To the right: 0.25mm notched bar, 'N0.25'.

Compacting tools have been designed for each type of fatigue bar. In all cases modified corners design has been adopted in order to avoid de-burring operations at the edges [8]. Figure 3 shows a modified corner on a polished FS bar cross section.

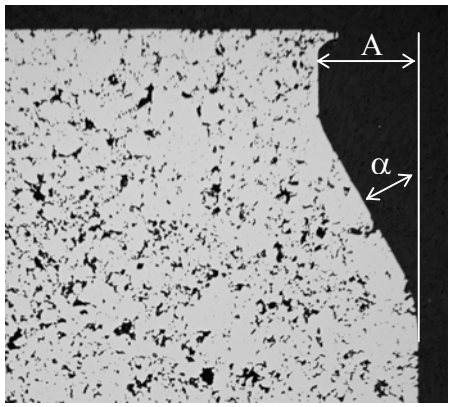


Figure 3. Modified corners for FS, N3, N0.9 and N0.25 bars [8]. A=0.3mm and α=30°.

Table 3 shows the Stress Concentration Factors of the notched bars, calculated according to a linear elastic material model [9].

Table 3. Stress Concentration Factor calculations (according to [9]) for the tested fatigue bars.

Fatigue bars	Notch Radius ρ [mm]	Stress Concentration Factor K_t
FS	-	-
N3	3	1.39
N0.9	0.9	1.79
N0.25	0.25	2.81

Fatigue testing

Four point bending fatigue tests have been run using the following parameters: load ratio R =-1 (i.e. mean stress equal to zero); frequency: 25-29 Hz; broken specimens stopped for 2.5% increased compliance; run out at 2 millions cycles; fatigue endurance [$\sigma_{A,50\%}$] was determined by staircase method, according to MPIF Standard No.56, 2001. 10 to 12 specimens have been included in the staircase calculations.
Figure 4 shows the test position in the bending fatigue machine used for all the type of bars –maximum stress on the die surface.

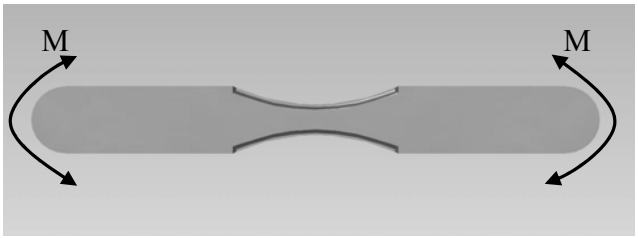


Figure 4. Loading mode adopted for all bars.

RESULTS

Microstructure

Ordinary light optical microscopy analysis (LOM) has been performed on polished and etched FS bars. Etching agent has been in both cases 4%Pikral solution (100ml Ethanol + 4g $C_6H_3N_3$).
Figure 5 shows the microstructures of D.AE and D.HP. In both cases the main characteristic is microstructural heterogeneity: a martensitic network surrounds the base powder particles and the Ni-rich austenitic islands. Retained austenite and spots of lower bainite can also be detected within the network. D.AE and D.HP show pearlitic (with spots of ferrite) and bainitic base powder particles respectively.

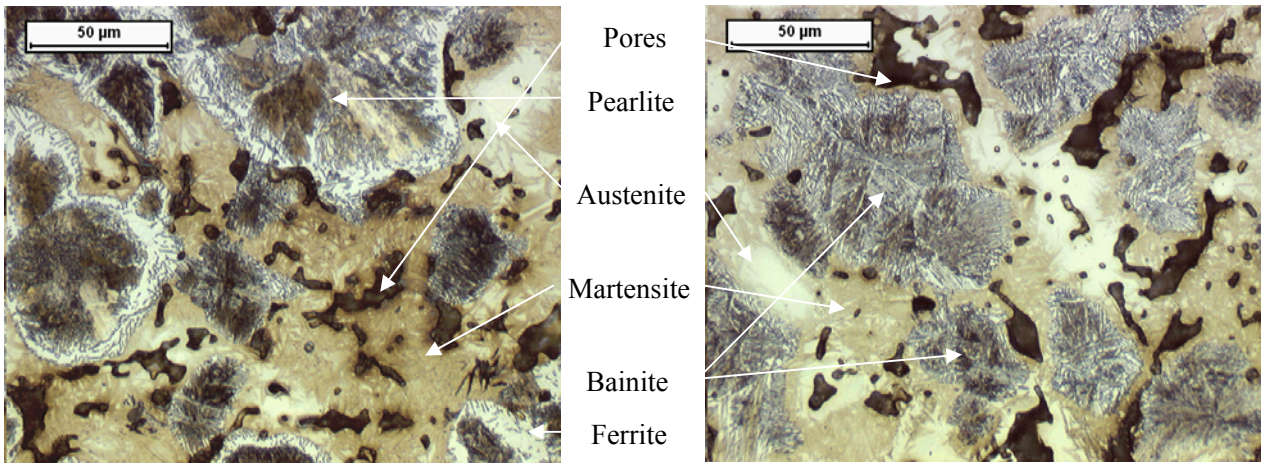


Figure 5. Microstructure of D.AE (to the Left) and D.HP (to the Right).

Fatigue tests results

Table 4 and Table 5 summarize the bending fatigue tests results respectively for material D.AE and D.HP. The scatter in the results in all cases was close to 5% of the $\sigma_{50\%}$ values.

Table 4. Fatigue tests results for all the series of D.AE.

Material	Notch Radius [mm]	$\sigma_{50\%}$ [MPa]
D.AE	-	275
	0.9	212
	0.25	196

Table 5. Fatigue tests results for all the series of D.HP.

Material	Notch Radius [mm]	$\sigma_{50\%}$ [MPa]
D.HP	-	322
	0.9	243
	0.25	241

DISCUSSION

Notch sensitivity of diffusion bonded materials

The maximum stress in the root of a notched bar is higher than the nominal stress (σ_{nom}) calculated from elementary beam theory. The theoretical maximum stress (σ_{max}) for a linear elastic material is calculated as $K_t * \sigma_{nom}$, where K_t is the stress concentrator factor. The tests performed show that σ_{max} exceeds the fatigue strength for un-notched test bars. This has already been shown for full dense materials as well as for PM steels [1, 2, 3, 4, 10].

The notch sensitivity factor, q, has been introduced in order to quantify a material's sensitivity to notches, i.e. to quantify the drop in fatigue performance due to the introduction of a notch in a component of a given material. The notch sensitivity factor is defined as:

$$q = \frac{K_f - 1}{K_t - 1} \qquad \text{(Equation 1)}$$

Where K_f is the fatigue notch factor, defined as the un-notched and notched fatigue strength ratio:

$$K_f = \frac{\sigma_{50\%}(Un-notched)}{\sigma_{50\%}(Notched)} \qquad \text{(Equation 2)}$$

q =1 corresponds to full notch sensitivity, and q=0 represents the hypothetical case of a material completely non-sensitive to notches.

Table 6 and Table 7 show the calculations of the notch sensitivity factors for material D.AE and D.HP respectively. Results for 3mm notch bars are from reference [4].

Table 6. Fatigue strength and notch sensitivity for as sintered D.AE. Results for 3mm notch bars are from reference [4].

Material	$\sigma_{50\%}$ [MPa]	Notch Radius ρ [mm]	Stress Concentration Factor K_t	Fatigue Notch Factor K_f	Notch Sensitivity Factor q
D.AE	275	-	1	-	-
	212	3	1.39	1.29	0.76
	196	0.9	1.79	1.40	0.51
	168	0.25	2.81	1.63	0.35

Table 7. Fatigue strength and notch sensitivity for as sintered D.HP. Results for 3mm notch bars are from reference [4].

Material	$\sigma_{50\%}$ [MPa]	Notch Radius ρ [mm]	Stress Concentration Factor K_t	Fatigue Notch Factor K_f	Notch Sensitivity Factor q
D.HP	322	-	-	-	-
	243	3	1.39	1.32	0.83
	241	0.9	1.79	1.33	0.42
	188	0.25	2.81	1.71	0.39

Notch sensitivity for both as sintered D.AE and D.HP decreases with increasing K_t. Thus, the notch sensitivity factor q varies with the asperity of the notch –see Figure 6. This could be related to the fact

that the probability of finding a microstructural defect acting as crack initiator is lower in a high-K_t test bar: for the fatigue bars geometries here considered, the more severe is the notch the smaller is the portion of material that due to the notch is under stress higher than the nominal.
Figure 7 shows the same fact in terms of increase of fatigue notch factor K_f with the asperity of the notch.

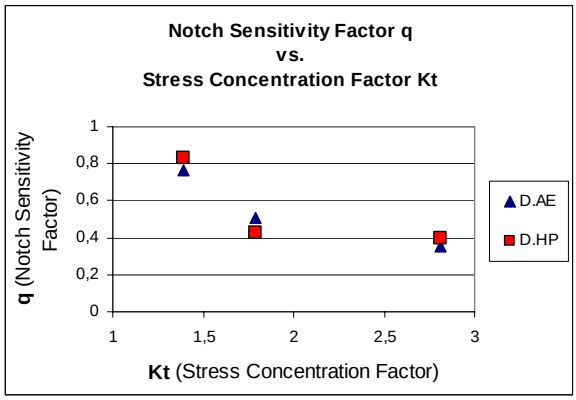


Figure 6. Dependence of notch sensitivity factor q on stress concentration factor K_t.

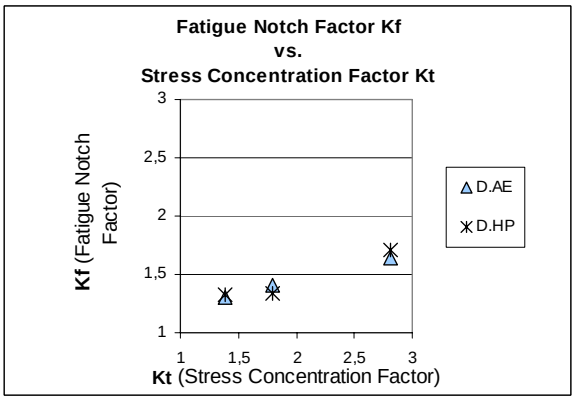


Figure 7. Dependence of fatigue notch factor K_f on stress concentration factor K_t.

Accordance with data presented in Literature is found for 2mm notched bars (drilled hole), tested under axial fatigue loading –see Table 8. Fatigue strength values were evaluated by linear regression for a 50% probability survival.

Material	Loading mode	Density [g/cm³]	Notch Radius ρ [mm]	Stress Concentration Factor K_t	Fatigue Notch Factor K_f	Notch Sensitivity Factor q	Reference
D.AE	Axial	7.07	2	2.8	1.52	0.29	[2]
D.HP	Axial	7.15	2	2.8	1.72	0.40	[3]
	Axial	7.29	2	2.8	1.74	0.41	[3]

Table 8. Notch sensitivity data from the Literature. All bars sintered at 1120°C/30', Carbon level close to 0.7%. 2mm holed obtained by drilling. Stress ratio R=-1.

Several theories relating notch sensitivity to material properties and notch geometry have been reviewed by Yao et al. [10].
The Neuber and the Peterson models have been applied to the q values determined in the fatigue testing. According to the Neuber model, the notch sensitivity index is influenced by an inner dimension, the elementary radius A. the notch sensitivity factor q can be expressed as:

$$q = \frac{1}{1+\sqrt{A/\rho}} \qquad \text{(Equation 3)}$$

where ρ is the notch root radius.
In the Peterson model, based on the assumption that the stress drops linearly near the notch, q is espressed in terms of notch radius ρ and the constant a, depending on material's strength and ductility:

$$q = \frac{1}{1+a/\rho} \qquad \text{(Equation 4)}$$

Table 9 and Table 10 summarize the Neuber and Peterson constants evaluation. The two models are not fitting with the experimental results obtained here.

Table 9. Fatigue strength and notch sensitivity for as sintered D.AE.

Material	bar	Notch Radius ρ [mm]	Notch Sensitivity Factor q	Neuber constant A	Peterson constant a
D.AE	N3	3	0.76	0.3	0.94
	N0.9	0.9	0.51	0.83	0.86
	N0.25	0.25	0.35	0.86	0.46

Table 10. Fatigue strength and notch sensitivity for as sintered D.HP.

Material	bar	Notch Radius ρ [mm]	Notch Sensitivity Factor Q	Neuber constant A	Peterson constant a
D.HP	N3	3	0.83	0.12	0.61
	N0.9	0.9	0.42	1.71	1.24
	N0.25	0.25	0.39	0.61	0.39

Microstructural characteristics of Diffusion bonded PM steels in as sintered state
On the basis of SEM Fractography and LOM-based crack walk analysis it has been previously reported that fatigue cracks in diffusion bonded materials at density close to 7.1g/cm^3 initiate in the base powder particles, while fully pre-alloyed materials show fatigue cracks originating in sinter necks [5, 6, 11]. In other words, diffusion bonded materials subjected to fatigue loading show low sensitivity to the local notch effect caused by pores facing the surface. In this paper notch sensitivity factors lower than 0,4 have been shown for stress concentration factors equal to 2,81. The explanation for this performance is that in diffusion bonded materials pores are within the martensitic areas: the Nickel and Copper-rich martensitic network plays the role of reinforcement for the sinter necks.

CONCLUSIONS

Bending fatigue test have been run on un-notched and 3, 0.9, 0.25mm notched bars of two diffusion bonded PM steels in as sintered conditions. Notched bars have been obtained by specifically designed compacting tools.
After compaction at 7.1g/cm^3 density level, sintering was performed at 1120°C (2050°F) in 90%N_2/10%H_2 atmosphere, cooling rate 0.8°C/s (1.4°F/s). Material D.AE is iron based, with 0.5%Mo, 4%Ni, 1.5%Cu diffusion bonded. Material D.HP is based on 0.5%Mo pre-alloyed iron based powder particles with 4%Ni and 2%Cu diffusion bonded.

- Notch sensitivity factors (q) decrease for both materials with increasing Stress Concentration factor (K_t).
- For material D.AE in 3, 0.9 and 0.25mm notch conditions q is equal to 0.76, 0.51 and 0.35 respectively. For material D.HP in 3, 0.9 and 0.25mm notch conditions q is equal to 0.83, 0.42 and 0.39 respectively.
- On a fatigue point of view, diffusion bonded materials in the as-sintered sate show microstructure already optimized with regards to sensitivity to notches and pores facing the surface.

REFERENCES

1. Cetin Morris Sonsino, “Elasto-plastiches Werkstoffverhalten und Schwingfestigkeit von neuentwickelten hochfesten Sinterstählen”, LBF-Bericht Nr.5590, Fraunhofer-Institut für Betriebsfestigkeit, Darmstadt, 1986, ISSN 0721-5320.
2. Cetin Morris Sonsino, “Stiegerung der Schwingfestigkeit von hochfesten Sinterstählen Teil1: Schwinfestigkeits-untersuchungen am Sinterstahl Fe-4%Ni-1,5%Cu-0,5%Mo mit variiertem Kohlenstoffgehalt, 1. Zwischenbericht”, LBF-Bericht Nr.6780/1, Fraunhofer-Institut für Betriebsfestigkeit, Darmstadt, 1990.
3. Ulf Engström, Klaus Lipp, Cetin Morris Sonsino, “Influence of Notches on Fatigue Strength of High Performance PM Materials”, PM2TEC 2001, New Orleans, USA, May 2001.

4. Hans Eriksson, “Influence of notches on Fatigue Behaviour of PM Steels”, Diploma work, Luleå University of Technology (Luleå, Sweden), Department of Applied Physics and Mechanical Engineering –Division of Engineering Materials. 2003:255 CIV, ISSN:1402-1617, ISRN: LTU-EX- -03/255- -SE, year 2003.
5. Luigi Alzati and Anders Bergmark, “Diffusion alloyed powders with high fatigue performance in the as-sintered state”, PM2004 World Conference, Vienna (Austria), 2004.
6. Luigi Alzati, Anders Bergmark and Jessica Andersson, “Fatigue Performance of PM Steels in as sintered state”, PMAI2005, Mumbai (India), 2005.
7. Anders Bergmark and Luigi Alzati, “Fatigue crack walk in Cu-Ni-Mo alloyed PM steel.” Special issue of Fatigue and Fracture of Engineering Materials and Structures (FFEMS), vol.28, No.1-2, 2005.
8. Anders Bergmark, Luigi Alzati, Ulrika Persson, “Fatigue Crack Initiation in PM Steel”, PM2TEC Powder Metallurgy World Congress 2002, MPIF, Orlando (USA).
9. Raymond J. Roark, Warren C. Young, “Formulas for Stress and Strain”, 5th Edition, McGraw-Hill International Editions, Singapore, 1986.
10. Yao Weixing, Xia Kaiquan, Gu Yi, “On the Fatigue Notch Factor, K_f,”, International Journal of Fatigue, Vol 17 No 4, pp 245-251, 1995.
11. Matsunaga H., Mårs O., Johannesson B. and Murakami Y., *“Statistical distribution of powder sizes and fatigue strength of powder metal”.* Higher Education Press, Fatigue '99: Proceedings of the Seventh International Fatigue Congress; Volume 3 (China), pp. 1817-1822, June 1999.