Modern Pre-alloyed PM Steels and Advanced Process Technology

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The development of new fields of application for powder metallurgy necessitates materials and process technologies which permit the economic production of high-load structural parts. High dynamic and static strength can be achieved by the use of pre-alloyed steel powders in combination with suitable heat treatment processes.
Direct sinter hardening of pre-alloyed steel powders containing nickel and molybdenum (mixed with copper and graphite) in special sinter furnaces with rapid cooling zone allows a martensitic of bainitic microstructure to be produced.

The paper illustrates the dynamic strength characteristics of two PM-steels MSP 1.5Mo (Fe-1,5%Mo, density 7,1g/cm³) and MSP 3.5Mo (Fe-3,5%Mo, density 7,5g/cm³), while the chemical composition as well as the sintering and heat treatment conditions were varied. MSP 1.5Mo is sintering in the γ-phase, whereas the alloy MSP 3.5Mo is sintering in the α-phase. The fatigue strength was investigated under axial loading and bending with the stress ratio R=0 and R=-1. Furthermore, instrumented impact bending tests were carried out to describe the impact behaviour quantitatively.

Although these sintered steels have differential strengthening mechanisms, for both steels there could be achieved high fatigue strengths by optimising the sintering and heat treatment conditions. The impact behaviour differs clearly. The dynamic properties of these materials after optimised heat treatment are explained as well as the finishing by a cutting process which often makes high demands on the cutting tools.

Introduction

P/M structural parts play an increasing role in the automotive industry. Especially applications in heavy-duty components like gears, pistons and connecting rods which are exposed high cycle dynamic loads can be realised with new developed materials [1]. For these new ranges of applications it is necessary to determine not only the static tensile properties, but also necessary to characterise the dynamic behaviour which can described by the fatigue and impact strength.

The dynamic properties of P/M parts are a complex phenomena which is influenced by numerous factors like pores, microstructure, heat treatment, etc. [2]. In several studies it has been proved that the porosity of a P/M part has the greatest influence [3, 4]. It can generally be assumed that raising the density leads to a more or less linear increase of the static mechanical properties, while the fatigue and impact strength increase exponentially.
In this paper the dynamic properties of two molybdenum-alloyed steels with different alloying contents, sintering and heat treatment conditions are represented. The aim of this investigation was to optimise these parameters and to give the designer useful characteristics of the new developed sintered steels. To complete the results there is a proposal for a cutting operation with new drilling tools as well a comparison between gears made from Mo-prealloyed sintered steel and conventional case hardening steel.

Materials and Microstructure

The investigations were carried out with two prealloyed steels with different molybdenum contents. Both materials are made by cold pressing (700 MPa) and single sintering technique. In Figure 1 data concerning the chemical composition, the sintering and heat treatment conditions, the density and the static mechanical properties are given.

Material	State	Sintering and heat treatment	Density [g/cm³]	Hardness HB2.5/187.5	$R_{p0.2}$ [MPa]	R_m [MPa]	A [%]	Micro-structure
MSP1.5Mo+0,8%C +0,8%MW+5%Car-bonyl iron	as-sintered	1250°C	7,18	182	530	705	0,65	Perlite
MSP1.5Mo+0,8%C +0,8%MW+ 5% MSP1.5Mo fine powder	as-sintered, quenched + tempered	1280°C 920°C/30min./H_2/oil 550°C/2h/N_2	7,14	316	991	1129	0,57	Martensite
MSP1.5Mo+0,5%C +0,8%MW+2%Cu	sintered, direct quen-ched+ tempered	1120°C/oil 500°C/1h/N_2	7,00	300	840	845	0,21	Martensite
MSP3.5Mo+ 0,8%MW	as-sintered (and case hardening)	1250°C	7,55	135	176	346	31,30	Ferrite
MSP3.5Mo-0,1%Nb +0,8%MW	as-sintered (and case hardening)	1290°C	7,59	141	203	377	29,17	Ferrite+ Martensite surface layer

Figure 1: Materials, manufacturing parameters, density and mechanical properties

The heat-treatable steel MSP 1.5Mo is suitable for connecting rods in the automotive industry. High dynamic properties are requested for this application. The alloying element molybdenum increases the tensile properties by solid solution hardening as well as the hardenability. Carbon causes an increase of the strength, microwax is used as lubricant. After sintering the material MSP 1.5Mo has a bainitic microstructure. A martensitic structure can be achieved by quenching directly after sintering or in an single hardening process.

MSP 1.5Mo is sintering in the γ-Phase and reaches thereby only a density of 7,18 g/cm³ in single sintering technique. The relatively low density leads to elongation values of 0,65 %. Very high strength properties can be achieved with the sintered steel MSP 1.5Mo.

MSP 3.5Mo can be applied for high-loaded gears and synchronisation gears because of it's high endurance and impact strength and it's good hardenability.

The sintered steel MSP 3.5Mo contents an increased molybdenum amount. Molybdenum encourages the formation of ferrite phase as shown in the phase-diagram Fe-Mo (figure 2). If the molybdenum content amounts to 3,5% by weight, only the ferrite phase exists during sintering at 1250 °-1280 °C in the walking beam furnace [5]. The sintering is promoted by the higher self-diffusion coefficient of Fe in the α-phase, which leads to an increasing density. The stabilisation of the α-phase is significantly reduced by carbon. Hence, the sintering atmosphere must not contain carbon. The microstructure consists ferrite with spherical fine pores (figure3). This leads to very high toughness values. MSP 3.5Mo was alloyed with phosphorus and niobium and afterward case hardened to increase the static and dynamic properties. In some specimens a higher molybdenum content of 4% was realised to improve the mechanical properties.

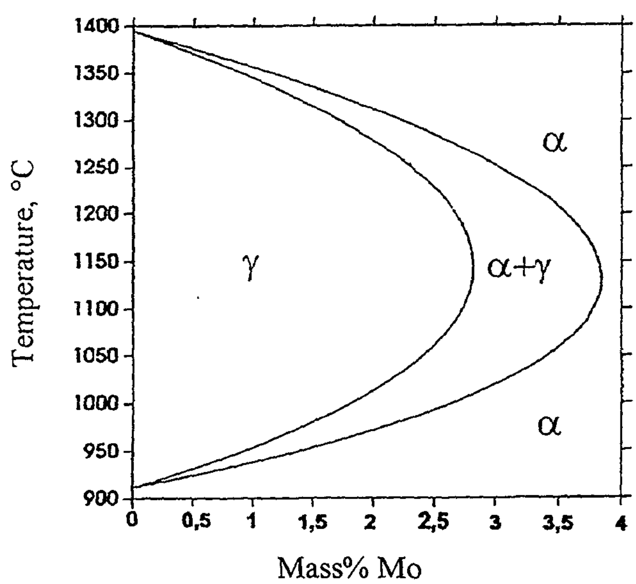


Figure 2: Phase diagram of Fe-Mo

Figure 3: Ferritic Microstructure of MSP 3.5Mo

Experimental Methods

The fatigue tests were carried out with unnotched rectangular specimens. After sintering and heat treatment each specimen was polished and cleaned. The fatigue tests were performed with axial loading and bending with the stress ratio R=-1 (alternating loading) and R=0 (pulsating loading) to determine the mean stress sensitivity. The fatigue testing followed the staircase method to assess the distribution in endurance strength at $2*10^6$ cycles. Each S-N curve was carried out with 30 specimens: 15 in the region of endurance limit (staircase method) and 15 in the region of finite life fatigue strength with varying horizons (Gaussian probability method). The S-N curves are illustrated in terms of stress amplitude σ_a and cycles to failure N.

Charpy impact tests on unnotched (DIN ISO 5754) and notched (DIN EN 10 045) samples were conducted with an instrumented pendulum in a temperature range from -150 °C to +150 °C. The impact hammer speed v_0 was 5423 mm/s and an available energy E= 300J. All specimens were oriented that the impact load was applied perpendicular to the direction of pressing to avoid the effect of density gradients. The results are presented in an impact energy transition curve. Furthermore two typical load-deflection curves of MSP 3.5Mo in as-sintered and case hardened state are given. First some predeterminations to characterise the impact behaviour at room temperature were carried out. Specimens made of MSP 3.5Mo in as-sintered state have to be notched, because they show such high a ductile behaviour and plastic deformation that they do not break at higher test temperatures.

The fracture surfaces of the specimens were observed by SEM.

Experimental results and discussion

Figure 4 and 5 present the endurance limits (P_s =50 %) under axial load and bending for the sintered steels MSP 1.5Mo and MSP 3.5Mo.

Generally the values for axial loading are lower than for bending. The stress gradient in the specimen depends on the kind of loading. In the case of axial loading the complete volume has to endure the maximum stress. In comparison to axial loading The stress gradients are much steeper under bending in comparison to axial loading, which leads to a reduced volume of maximum stresses. The smaller area of maximum stresses is, the higher is the probability to withstand the local stresses.

Moreover the figures 4 and 5 show that the stress ratio influences the endurance limit for both sintered steels. Pulsation load (R=0) decreases the fatigue strength due to the present mean stress.

The sintered steel MSP 1.5Mo was tested in various variations (chemical composition, sintering and heat treatment conditions).

In as-sintered state the alloy MSP 1.5Mo+0,8%C+5%Carbonyl shows low fatigue strength values under bending according to the low static mechanical properties like hardness, yield strength and tensile strength. It can be seen clearly that quenching and tempering increases the fatigue strength significantly, for axial loading as well as for bending. The endurance limit is raised as well by adding 5% fine powder of MSP 1.5Mo to improve the sintering activity.

Next it should be tested, if it is possible to reach the same properties by sintering the specimens in a continuous furnace at a temperature of 1120 °C with integrated rapid cooling instead of sintering in a walking beam furnace at a temperature of 1250-1280 °C. With the integrated cooling after the sintering process the manufacturing costs can be reduced significantly. As shown in table 1 and figure 5 the values of static and dynamic strength are lower than in as-sintered, single quenched and tempered state. It can be supposed that the main cause is the reduced density because of the lower sintering temperature. The specimens break without significant plastic deformation due to the low fracture elongation. Stress concentrations cannot be minimised. To reduce the liability to brittle fracture the carbon content was lowered to 0,5% instead of 0,8%.

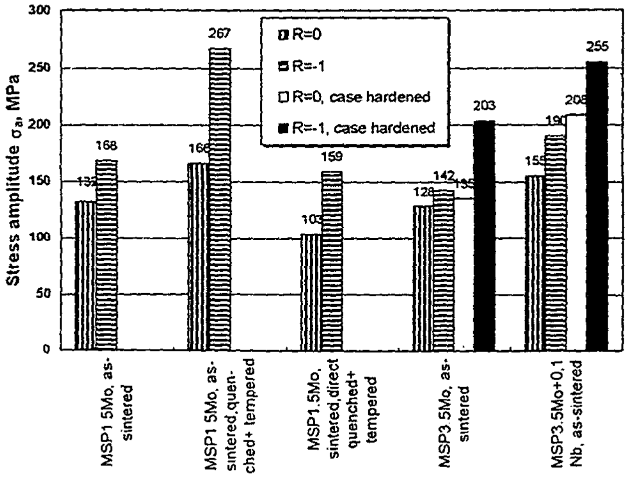


Figure 4: Endurance limit (P_s =50%) under axial loading for MSP 1.5Mo and MSP 3.5Mo

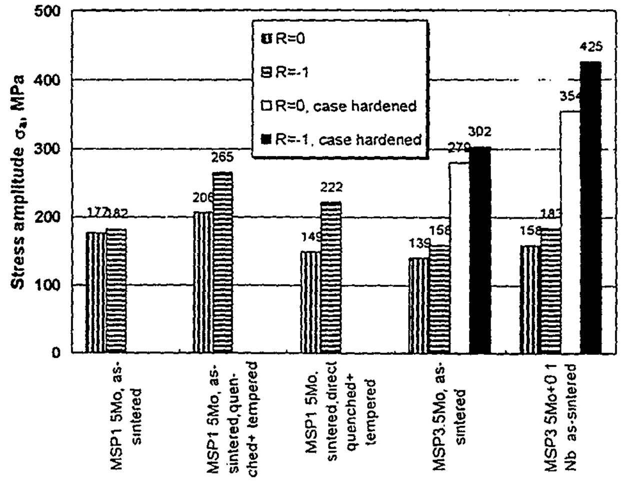


Figure 5: Endurance limit (P_s=50%) under bending for MSP 1.5Mo and MSP 3.5Mo

MSP 3.5Mo reaches only low fatigue strength values in as-sintered state under bending and axial load due to the ferritic microstructure which has also reduced static properties. An addition of 0.1% niobium and an increased molybdenum content lead to advanced values under bending and axial loading.

A significant improvement of the mechanical properties can only achieved by a case hardening or a carbonitriding. A fatigue strength under bending stress of 279 MPa (R=-1) and 302 MPa (R=0) can be reached by an optimisation of the case hardening parameters like for example austenitizing temperature, surface carbon content, specified depth of case-hardening EHT_{550} .The fatigue strength is nearly doubled by case hardening. The degrees of improvement caused by case hardening are higher for the MSP 3.5Mo alloyed with niobium. The S-N curve of MSP 1.5Mo+0,5%C+2%Cu under bending with the stress ratios R=0 and R=-1 is given in figure 6. Either the fatigue strength for finite life or the fatigue endurance limit is influenced by the stress ratio and the main stress.

Figure 7 shows the S-N curve of the MSP 3.5Mo in case hardened state under axial loading. Here the improvement caused by case hardening is relatively small compared to bending. The whole specimen volume must endure the stresses under axial loading. Under bending the stress gradient is deeper, only the area on the surface has to withstand the maximum stresses. The surface area has the highest carbon content and hardness due to the case hardening. Therefore the effect of case hardening is more significant under bending.

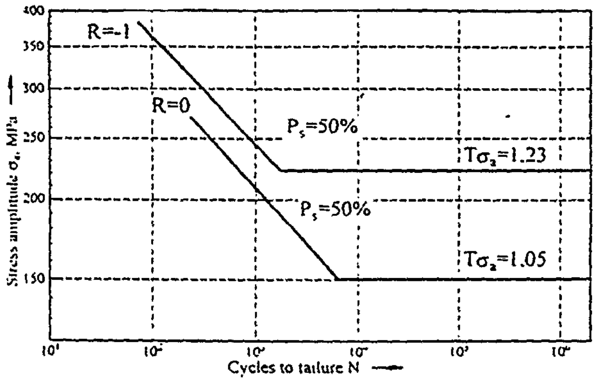


Figure 6: S-N curve of MSP1.5Mo+0,5%C+2%Cu; sintered, direct quenched and tempered, **bending**

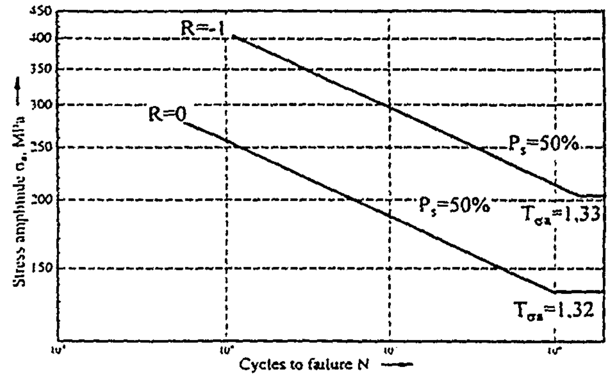


Figure 7: S-N curve of MSP 3.5Mo, case hardened, **axial loading**

Impact curves at room temperature for the material MSP 3.5 Mo in as-sintered state and case hardened are shown in figure 8. The MSP 3.5 Mo in as-sintered state behaves very ductile which can be explained by the high density. The deflection at onset of unstable crack propagation is very high, stable crack propagation has taken place in as-sintered state. The deflection of fracture decreases and the maximum load increases with increasing hardness caused by case hardening. Here no stable crack propagation was possible.

Figure 9 presents different impact energy transition curves of MSP 3.5Mo, MSP 3.5Mo+0,1%Nb and MSP 1.5 Mo+0,8%C+5%MSP 1.5Mo fine powder.

MSP 3.5Mo and MSP 3.5Mo+0,1%Nb in as-sintered were tested with notched test bars, because it was not possible to break them at higher temperatures in unnotched state. MSP 3.5Mo and MSP 3.5Mo+0,1%Nb in as-sintered state show a clear transition from ductile to brittle. At elevated temperatures the specimens show a strong plastic deformation and high impact energies (approximately 25 J). Fracture surface observations in the SEM of the MSP 3.5Mo in as-sintered state indicate that elevated temperatures cause ductile fracture (figure 10). Here the dimples due to plastic deformation can be clearly observed.

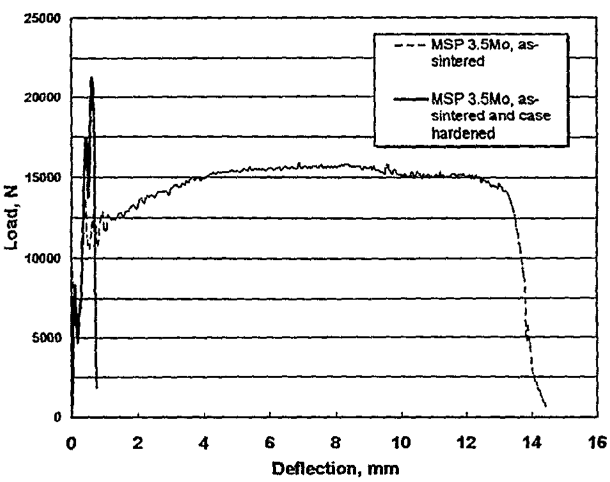


Figure 8: Impact curves for MSP 3.5Mo in as-sintered and case hardened state at room temperature

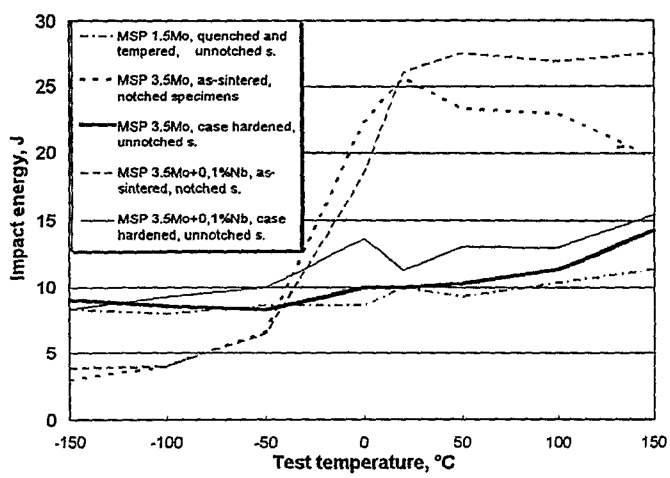


Figure 9: Impact strength transition curves

MSP 3.5Mo has spherical pores due to the activated sintering in the α-phase which lead to an decreasing of the stress concentrations around the pores. The behaviour of the material will be similar to that of cast and forged materials: the Charpy energy at high temperatures increases. At low test temperatures the crack will propagate promptly [6]. The fracture appearance changes to transgranular cleavage fracture (figure10). The plain surfaces of fracture and the pores are clear to study. The complete change in fracture character from ductile to brittle did not occur suddenly. Samples tested at transition temperature have mixed fracture mode side by side (cleavage and dimples).

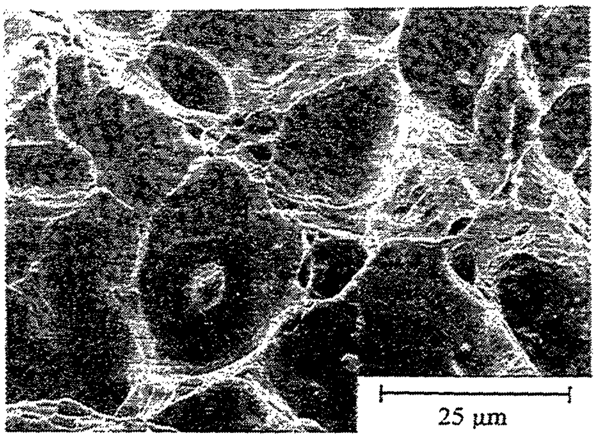


Figure 10a: Fracture surface of MSP 3.5Mo in as-sintered state at +150 °C Magnification: 1000:1

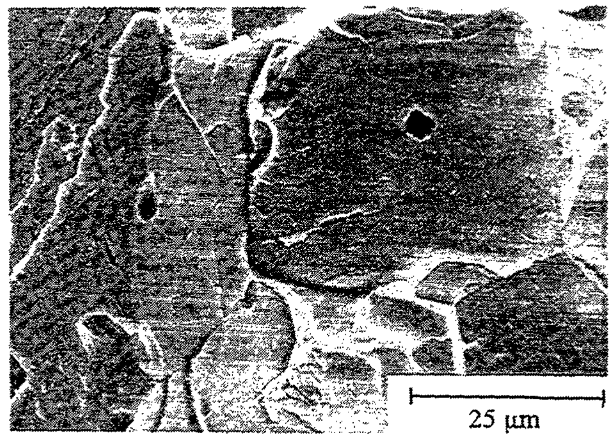


Figure 10b: Fracture surface of MSP 3.5Mo in as-sintered state at -150 °C Magnification: 1000:1

Case hardening reduces the impact strength and increases the brittleness of the materials due to surface hardening. The martensitic microstructure exhibits only a very low plastic deformability. The critical crack condition for crack initiation and propagation is immediately reached. The transitions curves show no clear transition from ductile to brittle.

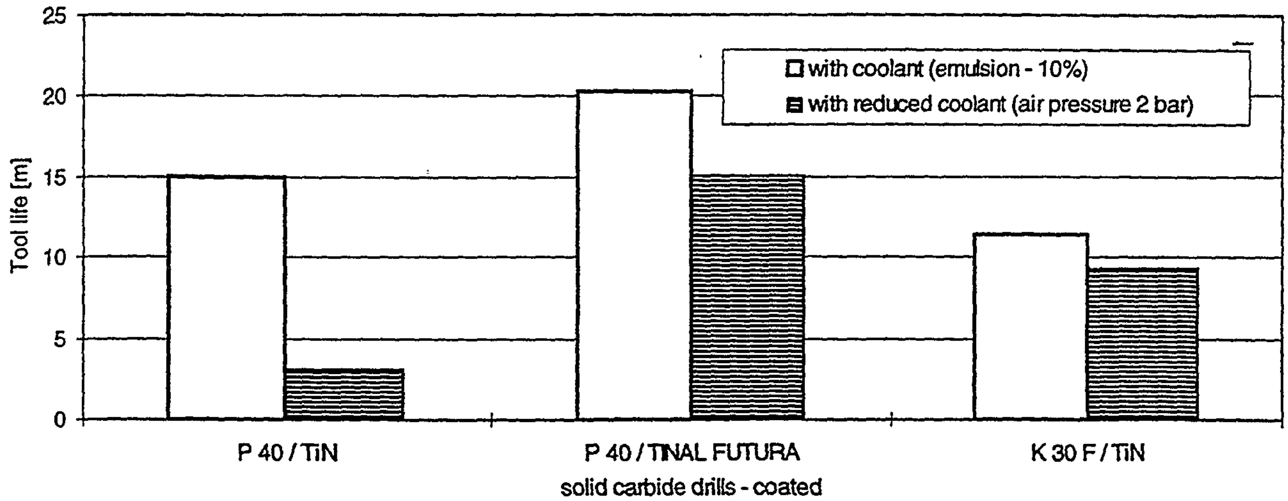
MSP 1.5Mo+0,8%C+5%MSP1.5Mo fine powder reacts very brittle. This material gave the most sluggish transition. The impact behaviour is connected with the low plastic deformability of the tempered martensitic matrix of MSP 1.5Mo+0,8%C+5%MSP1.5Mo fine powder. Once a crack is initiated, there is practically no ductile stable crack extension and fast fracture occurs.

Mechanical finishing

Although the main benefit of the PM technology is the direct, near-net shape production of low-cost compacts, almost 30% of the parts for the automobile industry are mechanically finished; 55% of all sintering plants carry out this work in their own plant [9].

If different materials such as compact steel and sinter steel are compared with respect to their machinability, tool and cutting parameters are frequently selected as constants. This can lead to distirted results, e.g. in the case of drilling, as here the tool - defined by material, geometry and coating - has to be adapted to the machining task.

A TINAL-coated solid drill, for example, is suitable for drilling rapidly cooled parts of prealloyed powders containing 1.5% molybdenum (Figure 11) [10].



Workpiece material:		Cutting parameters:	
MSP1.5Mo+2%Cu+0,6%C+0,8%MW (without MnS)		cutting speed:	vc = 40 U/min
makro hardness:	200 HV 30	feed rate:	f = 0,17 mm/U
mikro hardness:	370 HV 0.05	drill diameter:	d = 6,8 mm
tensile strenght:	730 N/mm²	depth:	t = 20 mm
test part dimensions:	Ø 65 mm x 30 mm		
sintered density:	7,06 g/cm³ (600 MPa)		
sinter temperatur:	1120°C with rapid cooling (T ~ 3°C/s)		

Figure 11: Drilling of MSP 1.5 Mo with solid carbide tools

With the coated solid carbide drill a high tool life of more than 20 m can be reached drilling this high strength sintered steel (MSP 1.5 Mo). Responsible for tool wear is the high micro hardness of 370 HV 0.05.

Comparison of sintered and conventional gears

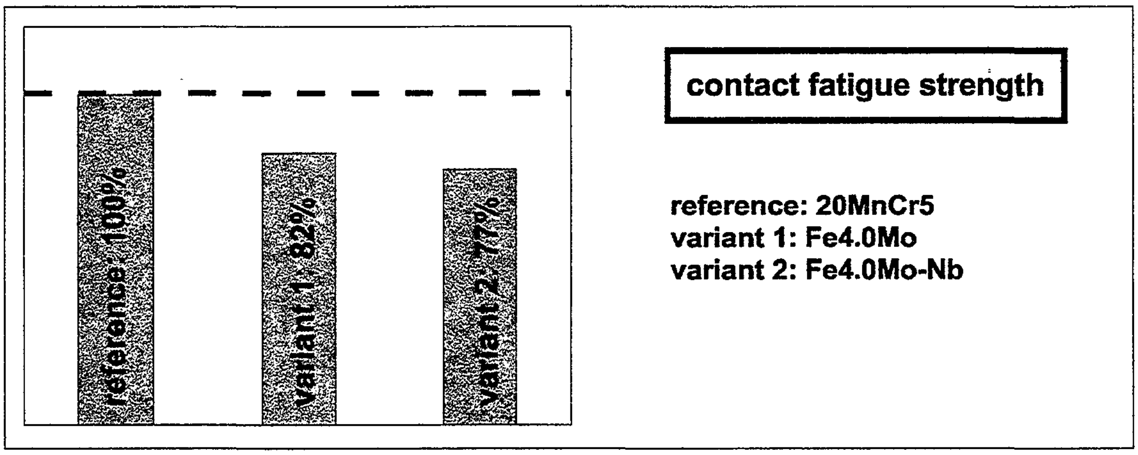
The economical and environmentally compatible PM process is a near-net-shape forming method that can open up new and innovative application potentials for powder metallurgy in the transportation sector. It is a suitable production method for automobile gears that are subject to high loads. This application represents an immediate means of raising the portion of PM components per car from the current level of approx. 5 kg.

PM components based on MSP 3.5 Mo can be manufactured with conventional production machines. This material is therefore expected to open up entirely new applications for finished precision parts; these can constitute an alternative to machined castings and steel components. In initial trials conducted as part of a preliminary study for a joint project under the auspices of the Federal Ministry of Education and Research, MSP 3.5 Mo has shown that PM gears with densities > 7.50 g/cm³ can be manufactured by single sintering with wear and endurance strength properties that are comparable to those obtained with low-alloyed compact steels. This is why the forenamed current joint project for the transportation sector is focusing on the development of new PM materials, in particular for manufacturing durable spur-toothed gears for the gearboxes.

The project spans the period 1995 to 1999 and has the following participants:

- Mannesmann Demag AG Hüttentechnik Meer, Mönchengladbach
  - Institute of Ferrous Metallurgy (IEHK) at Aachen Technical Univesity
  - Machine Tool and Management Theory Laboratory (WZ) at Aachen Technical University
- Sinterstahl GmbH, Füssen
- ZF Friedrichshafen AG, Friedrichshafen
- VW AG, Wolfsburg

In figure 12 there is a summary of latest results concerning the above mentioned development project.



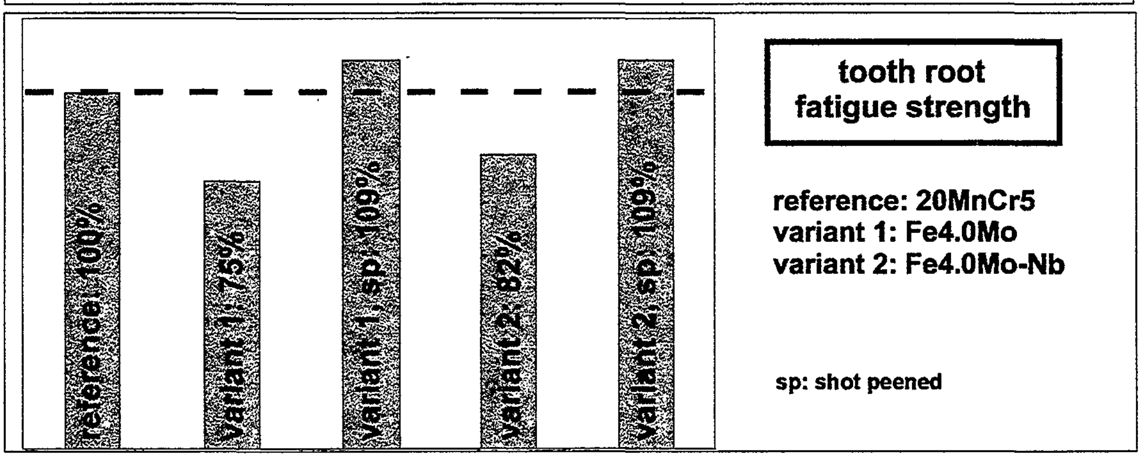


Figure 12: Component tests with sintered rollers and gears in comparison with solid case hardening steel (surface hardness: 60 HRC)

To determine that tooth root fatigue, the gear variants were subjected to a dynamic load on a resonance pulsator. The lower force F_u was set at 3000 N. The tooth pair was clamped between two jaws over four teeth, so that the line of dynamic effect lies in the vicinity of the outer point of action of the individual teeth.
A direct comparison as regards the continuously endurable pulsator forces can be made in this case between the machined reference variant and the sintered variants because the same tooth geometries were used (Gear data: $M_n = 3.5$ mm; $b = 20$ mm; $z = 25$; $\alpha = 20°$; $\beta = 0°$).

To investigate the contact fatigue, operating tests were conducted on an FVA-type mechanical torsion test rig with a distance between centers of 91.5 mm. In the test gear, the sintered test pinion was paired with a sintered mating gear. The wear criterion was the occurrence of pitting on the driving gear (test pinion).

The comparison between solid steel (20 MnCr 5) and the sintered variants shows, that the gap is very small and as far as the tooth roof fatigue is concerned shot peening increases the strength up to 109 % compared with the reference.

The reasons for this improvement in the load capacities lie in the reduced cracking tendency in the near-surface area, thanks to the thorough compaction of the near-surface layer, and to the creation of high compressive internal stresses in the near-surface area of the tooth flank.

Conclusions

For both sintered steels MSP1.5Mo and MSP3.5Mo the target was reached to develop materials for dynamically high-loaded applications. This study shows the different between both material concepts due to the different sintering behaviour either in α- or γ-phase and material composition.

The sintered steel MSP 1.5Mo achieves good fatigue strength values by high-temperature sintering with subsequent quenching and tempering. A sintering at 1120 °C with direct quenching to save costs doesn't lead to satisfying values MSP 1.5Mo shows a very brittle impact behaviour caused by the relatively low density and the martensitic microstructure, which exhibits only a low plastic deformability.

MSP 3.5Mo sintered in α-phase which requires a very low carbon content reacts very ductile due to it's high density and to the ferritc microstructure, but the static and dynamic strength values are very low. With a proper alloying elements and s subsequent case hardening it is possible to double the dynamic strength values. This is connected with a decrease in toughness.

The use of the prealloyed steel powder MSP 3.5Mo concentrates on PM components that must satisfy very stringent requirements as regards fatigue strength under reversed bending stresses and surface hardness, such as spur-toothed gears, synchroniser hubs and synchroniser rings for automobile gearboxes.

Acknowledgement

The investigations described in the present paper were conducted as part of a preliminary study for the forenamed project, sponsored by the Federal Ministry of Education and Research (Project No. 03N3024), at the Machine Tool and Management Theory Laboratory (WZL) at Aachen Technical University. The presentation also cites initial results from the joint project. We wish to thank the Federal Ministry of Education and Research (BMBF) for its financial support.

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