Alternative Mechanical Milling routes for grain-refinement of conventional High-Speed Steel powder for later consolidation by SPS

[1,3]H. Zoz , [2]K. Ameyama, [1,2] S. Umekawa, [1] H. Ren and [3] D. Jaramillo V.
[1] Zoz GmbH, D-57482 Wenden, Germany
[2] Ritsumeikan University, 525-8577 Kusatsu, Japan
[3] ESIQIE, National Polytechnic Institute, Mexico City, DF 07300, Mexico

Abstract:

High-speed steel is the most superior tool steel in terms of wear resistance at high temperature.
In order to further improve materials properties, conventional HSS-powder (<76µm) has been mechanically milled by a Planetary Ball Mill (medium kinetic system) and alternatively by a Simoloyer (high kinetic system) in order to reduce particle size and in particular the grain size of the powder material. Consolidation has been done by Spark Plasma Sintering in order to maintain a fine structure of the material.
In the Planetary Ball Mill, 25 g of HSS-powder was milled for 720 ks (200h) and resulted in a particle size of <15µm at an average grain size of 10nm. Similar results in the Simoloyer were achieved after 72 ks under higher powder load.
The SPS-sintered parts were investigated by hardness, the characterization of the powder is given by XRD, SEM and laser diffraction.

1. Introduction

High-speed steel is well known to be the most superior tool steel with high wear resistance property at high temperature. Main applications are cutting tools, dies etc. Some examples are given in Figure 1.

Figure 1: tools by HS-Steel, a: cutting tool, b: punch, c: die.

In an attempt of using powder metallurgy to improve the properties of conventional HSS, made by the melting method, the usually heterogeneous dispersion of carbides is a promising area of improvement. If the carbides can be distributed finer and more homogeneous where powder metallurgy can control such microstructure, better mechanical properties and workability can be expected.
Furthermore, the powder metallurgy route enables the material to be high respectively higher alloyed, so that again the mechanical properties of the material can be improved to higher grade.

Mechanical milling (MM) is one of the high strain powder metallurgy (HS-PM) processes. Because high strain is introduced from multi directions into powder-material in the MM process, super fine grain powder in non-equilibrium state can be obtained and this can lead to nano-crystalline or amorphous structure. By a novel process, we can control the microstructure to an ultra fine grain structure by using the formation of a new phase, the phase transformation, and the recovery and recrystallization when the MM powder is consolidated. The ultra-fine grain materials can be consolidated at low temperature and low pressure because of the superplastic deformation. The fine grain materials are more superior in the mechanical properties such as tensile strength etc. [1].
Fig. 2 shows the TEM-investigation of MM-HSS-powder milled for 720 ks in an ordinary ballmill. The bright field image is indicating nano-grains with grain-sizes of 10-20 nm of bcc structure where usually

this material consists of a phase-mixture of fcc and bcc structure and after MM an entire change to bcc structure could be noticed.

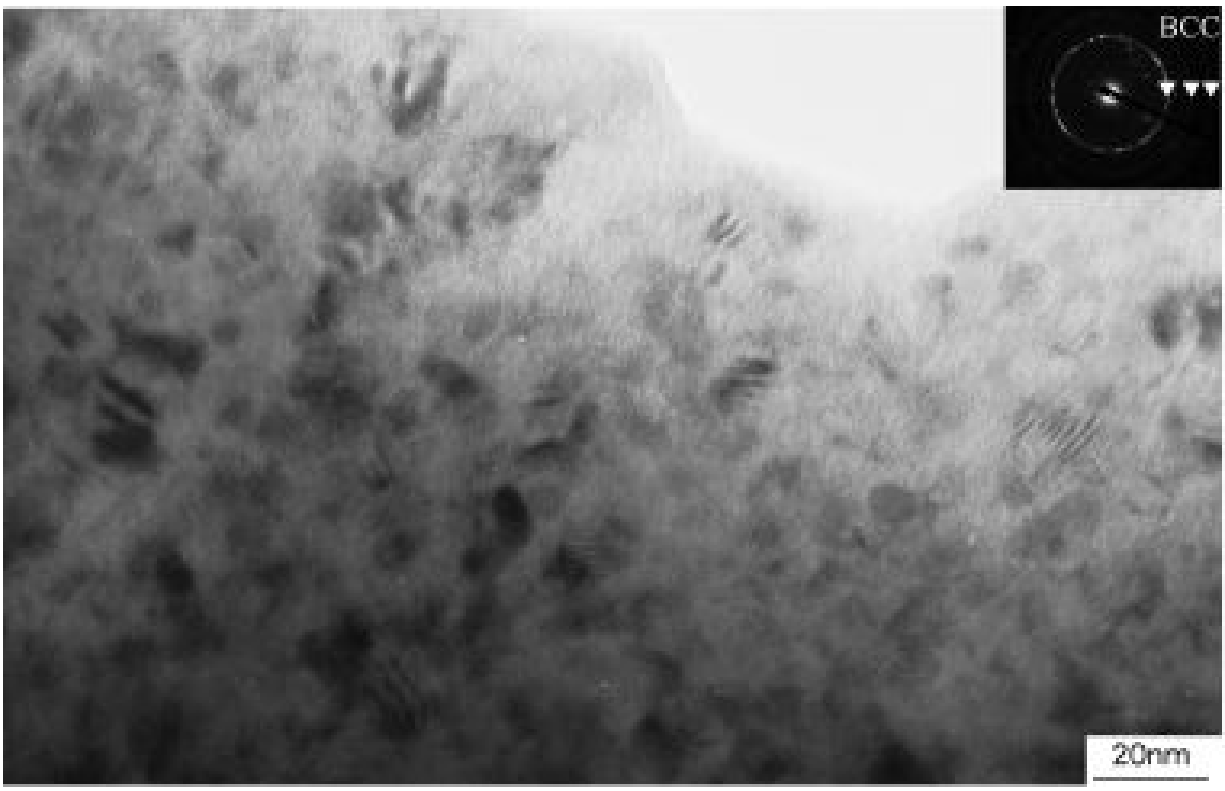


Figure 2: TEM bright field analysis of MM-HSS powder identified in table 1.

Process parameters of HSS powder in low kinetic MM process	
milling device	planetary ballmill Fritsch P-5
milling vessel	self-made, air-cooling-bars (SKD11, 500 cc)
grinding media	high alloyed steel, SUJ2, 9.8 mm
grinding media load	100 pc (360 g)
starting powder	HSS powder
starting powder load	25 g
powder/ball weight ratio	1 : 14.4
atmosphere	Argon after loading
rotational speed	250 rpm
MM time	720 ks (200h)
milling temperature	< 50 °C (vessel outside)
powder yield	approx. 10 g >> 40 %

Table 1: process parameters of HSS-powder in low kinetic MM-process

Table 1 gives the parameters that have been used in earlier work [2] to obtain the material shown in figure 2. The processing time at 720 ks which refers to more than 8 days is quite long and in fact too long to be realistically considered for a commercial production scale up. This general problem is due to the relatively low maximum relative velocity of grinding media in the planetary ball mill at about 5 m/s [3-4]. Also technically a process based on the planetary ballmill can not be scaled up to mass-production since here the entire container is performing the milling action by moving. This would mean tremendous energy losses and is simply a technical barrier. E.g. it is impossible to imagine to move containers with a total weight of e.g. 500 kg in this kind of kinetic. And since the unloading of the powder can only be carried out manually and this at powder yields of usually below 50 %, this also seem to be strict barriers.

Therefore in this work, the low-kinetic planetary ballmill process is compared with the processing in a high kinetic horizontal rotary ballmill (Zoz-Simoloyer) [5] which supplies 2-3 times higher kinetic and is of realistic scalability [6-8]. Both techniques are applied for the MM of HS-steel powder.

2. Experimental

2.1 Experimental, HS-steel powder

Comparatively, the MM-process was applied in both, low-kinetic and high-kinetic processing devices (see chapter 2.2 and 2.3) to the high-speed steel Fe-6W-5Mo-4Cr-3V-9Co-1.25C (mass%) where the detailed composition is given in table 2:

processed HS-steel powder in low- and high-kinetic MM							
element	C	W	Mo	Cr	V	Co	Fe
mass%	1.25	6.04	5.03	3.95	2.94	7.89	bal.

Table 2: detailed composition of HSS-powder Fe-6W-5Mo-4Cr-3V-9Co-1.25C

The powder was made by gas atomizing at Kobe Steel and is defined as KHA30 (Kobe High Atomel). The particle size of the used grade is < 900µm.
Since the planetary ballmill requires smaller starting powder size in order to obtain a homogeneous microstructure, the powder had been classified by sieving to < 75 µm (200 mesh sieve). This was not expected to be necessary for the Simoloyer and therefore not done in this case (table 3).

starting powder particle size for low- and high-kinetic MM			
low-kinetic	Planetary ballmill	< 75 µm	by 200 mesh sieve
high kinetic	Simoloyer	< 900 µm	as received

Table 3: starting powder particle size for low- and high-kinetic MM

2.2 Experimental, MM-process in Planetary ballmill

The principle of the planetary ball mill is, that the vessel filled with grinding balls and powders is rotated in a planetary orbit so the milling balls impact each other mostly by sheer and friction effects and partly also by collision with the vessel wall and partly of each other. Figure 3a shows a real image of a low kinetic mill in operation where the effect of shear and friction in MM is visualized. It is important to notice, that the given picture is not obtained from a planetary ballmill since this is not available and probably almost impossible (vessel moving in orbit). Therefore for demonstration, the available image of the ordinary laboratory ballmill (Zoz-RM1) has been chosen. Figure 3b shows a picture of the used planetary ballmill Fritsch P-5.

Figure 3a: shear & friction effects in low-kinetic MM (Zoz-RM1)

Figure 3b: planetary ballmill Fritsch P-5

In this device, MM was carried out for 180 ks and for 720ks and the process-parameters are given in table 4. For processing, the vessel of the mill is removed and carried into a glove-box in order to load and maintain the Ar-atmosphere in the milling-vessel. Then it was returned to the mill for the MM. Problematic is the control of the milling temperature since there is no cooling system available. The only way is to cool the environment (e.g. to place the mill into a fridge). An improvement is therefore a vessel

with larger surface for a better temperature exchange with the environment. By using the self-made PBM-container shown in figure 4, it has been possible to maintain an outside vessel temperature > 50°C at least.

Process parameters of HSS powder in low kinetic MM process	
milling device	planetary ballmill Fritsch P-5
milling vessel	self-made, air-cooling-bars (SKD11, 500 cc)
grinding media	high alloyed steel, SUJ2, 9.8 mm
grinding media load	100 pc (360 g)
starting powder	HSS powder
starting powder load	25 g
powder/ball weight ratio	1 : 14.4
atmosphere	Argon after loading
rotational speed	250 rpm
MM time I + II	180, 720 ks (50, 200h)
milling temperature	< 50 °C (vessel outside)
average powder yield	approx. 10 g >> 40 %

Figure 4: planetary ballmill container, self-made

Table 4: process parameters of HSS-powder in low kinetic MM-process

After the MM-time of 180 res. 720 ks, the vessel was transferred into a glove-box and powder and balls were unloaded onto a sieve in order to receive the powder only. Here it is of course very difficult to remove all of the powder, in particular because some of the material is sticking at the inner vessel-wall and at the milling balls. The received powder yield was in total only about 10 g which refers to 40 % yield approx.

2.3 Experimental, MM-process in the Simoloyer

The Simoloyer is a horizontal high energy ball mill and is known from academic as well as industrial applications in mechanical alloying (MA) [4, 9], high energy milling (HEM) [10] and reactive milling (RM) [11]. These devices supply the highest relative velocity of grinding media, which leads to a high level of kinetic energy transfer, an intensive grinding effect and short processing times. The contamination of the processed powders by the milling tools is naturally lower since the process is based on the collision of grinding media rather than on shear and friction interaction of the same which usually leads to higher abrasion. Since the grinding media is accelerated by a horizontally arranged rotor inside the grinding vessel, these devices do not have to move unnecessarily any large masses like e.g. the entire chamber/mill in case of vibration- or planetary ballmills.
The systems are available in large scale of several hundred liters volume and are economically and ecologically favorable in particular since in most cases they can be operated semi-automatically if they are combined with a continuous or semi-continuous (auto-batch) powder separation system. Atmosphere and cooling seems non-problematic since these mills can be operated, loaded and unloaded under vacuum or inert gas and are equipped with efficient cooling or cooling and heating systems.

Figure 5a: collision effects in high-kinetic MM (Simoloyer)

Figure 5b: horizontal high energy ballmill (Simoloyer CM01-2l) with air-lock for loading, operation and unloading under vacuum and/or inert-gas

Figure 5a shows a real image of a horizontal high energy ball mill in operation where the effect of collision in MM is visualized and the working principle can be imagined where the rotor is the tool to transfer the kinetic energy into the grinding media and the grinding media transfers into the powder material.
Figure 5b shows the same device (but with a different grinding unit, Simoloyer CM01-2l) that can be placed on a table next to the process controlling computer which is operated with water cooling or heating at rotation frequencies up to 1800 rpm. This laboratory-scale size has been used for the MM at high kinetic and was carried out in total for 72 ks (10 times shorter than in case of the planetary ballmill). The process-parameters are given in table 5.

Process parameters of HSS powder in high kinetic MM process	
milling device	Simoloyer CM01, 2.7 kW,
operating software	Maltoz 3.1
grinding unit	W01-2l (2 liter, water-cooled)
grinding media	Chromium steel, 100Cr6, 5 mm, 2 kg
starting powder	HSS powder
starting powder load	200 g
powder/ball weight ratio	1:10
atmosphere	Argon, preceding evacuation
rotational speed operation	1300 rpm
rotational speed discharging	cycle operation 900/1300 rpm
MM time I - IV	3.6, 18, 36, 72 ks (1, 5, 10, 20h)
milling temperature	< 25 °C (vessel inside by Maltoz)
feeding system	standard air-lock DN40-KF
discharging/separation	draingrating Ask-01
average powder yield	approx. 100 g >> 100 %

Table 5: process parameters of HSS-powder in high kinetic MM-process

Since for all different MM-times, the largest available amount of powder (for consolidation) is wanted, the sampling-unit was not used. For each time of 3.6, 18, 36 and 72 ks, a separate MM-process was operated. For loading the starting powder, first the mill that must be in charging/operation position is evacuated to around 10^{-4} hPa and then flooded with Argon. The powder, that was loaded inside a glove-box into a powder-container, was transferred by the air-lock into the vessel at the same time when the vacuum was flooded since then the gas flow can even be used for the powder transportation.
After the adjusted MM-time elapsed, the grinding unit is tuned into discharging position and the same air-lock (after cleaning) was used for unloading the powder where this time first the powder container is evacuated via the air-lock. In that way, the gas-flow supported the unloading (a little) and the powder was discharged completely (grinding media remains in vessel).

Unloading is computerized and automatically operated. In order to increase the powder yield and to decrease the discharging time, Cycle Operation has been applied which means during discharging, the rotational speed of the rotor is changed in a special frequency in the range from here 900-1300 rpm which has been proven to tremendously increase the powder yield of materials that tend to stick and agglomerate under MM to the milling tools and to each other [4, 9-11]. By this method a complete powder yield could be obtained. Since between the each of the different MM-operations, the system has never been opened and since a little amount of powder always remains in the system e.g. sticking to grinding balls and vessel, the powder yield varied between 95 and 110 % after a Discharging-Cycle of 15 min at 900/1300 rpm. Both of the cooling systems (vessel and pre-seal-unit) were constantly operated and the computerized measurement of the inner-vessel surface indicated temperatures lower than 25°C.

2.4 Experimental, consolidation by SPS

After characterizing, which will be given in chapter 3 (results), the MM powders were to be consolidated. Since in this work, next to a homogeneous carbide dispersion we want to take care for grain-refinement in an ultra-fine microstructure of the composite, the Electrical Discharge (Spark) Plasma Sintering (SPS) technique was applied, because it is expected, that by this the favoured microstructure can be maintained also after consolidation [12].

consolidation parameters of MM HSS-powders	
sintering device	SPS-510L
loaded powder	5.0 g
sintering temperature	1223 K
heating rate	1.76 K/sec
sintering time	0.3 ks (5 min)
sintering pressure	90 MPa
sintering condition	vacuum, <15Pa
dimension of die/sample	15 mm diameter

Table 6: consolidation parameters of MM HSS-powder

Figure 6: Electrical Discharge (Spark) Plasma Sintering device

Figure 7: SPS-compact, d= 15 mm

We used an SPS-510L (by Sumitomo Coal mining Co., Ltd.) which is shown in figure 6 and the consolidation parameters are given in table 6, to compact the MM-powders to small cylinders in the diameter of 15 mm (see figure 7). The applied sintering temperature was 1223K, reached at a heating rate 1.76K/sec. The holding/sintering time was 0.3ks at 90 MPa under vacuum. The achieved samples were the investigated for mechanical properties and are compared with conventional HS-steel manufactured by HIP or forging which will be given in chapter 3 (results).

3. Results

3.1 Comparison of powder yield, particle size and shape

Since the powder yield is important for any economic consideration of the here discussed material, and since the development of the particle size versus the milling time can carefully be used as an early identification of the process-efficiency, this data is compared as follows:
The graph as given in figure 8 summarizes the powder yield results at the determined processing time and is visualizing and confirming what has been expected, which is that the planetary ball mill leads to a poor powder yield below 50 % and the Simoloyer to a high yield around 100 %. In absolute numbers, in the Simoloyer approximately 200 g and in the planetary ballmill approximately 10 g are received, which is 20 times less.

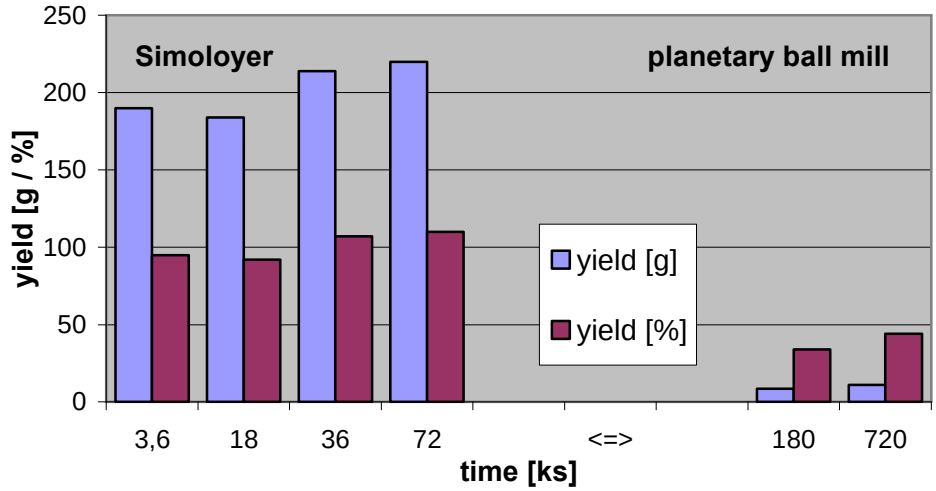
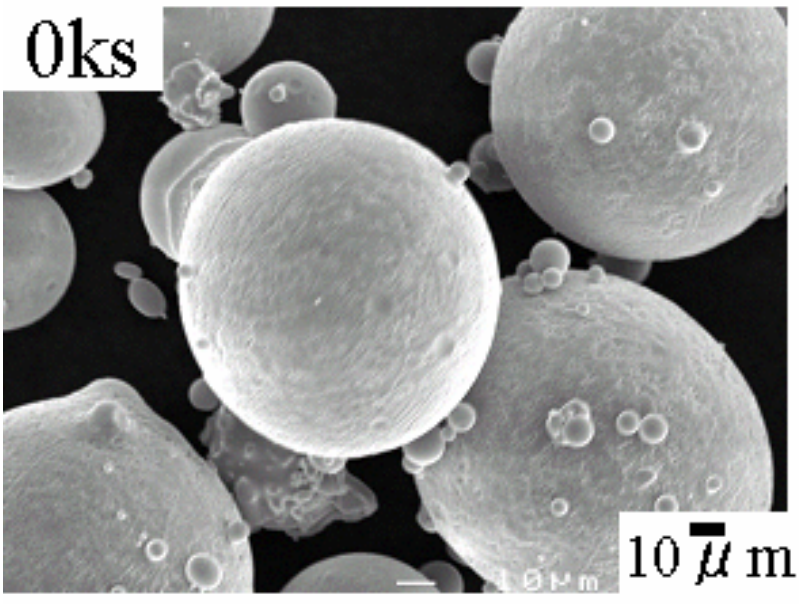
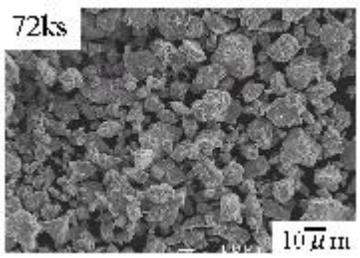


Figure 8: powder yield results & processing time after high- and low kinetic MM

Figures 9 and 10 compare the particle size development (estimating by SEM) and also the shape change but shape will be commented later. We used a JEOL JSM-6400, later for X-ray diffraction we use Rigaku RB-200BX diffractometer using monochromatic CuKa radiation.
Figure 9 shows on the left hand side the SEM of the starting powder for the MM at high kinetic which has been used as received and the given data here was < 900 µm. The image confirms a gas atomized powder in spherical shape with some satellite-effects in an average particle diameter of 120 µm. After 72 ks, which is given in figure 9 on the right hand side, size and shape dramatically changed. The average size is now estimated to be about 15 µm and the shape is fissured and flattened.



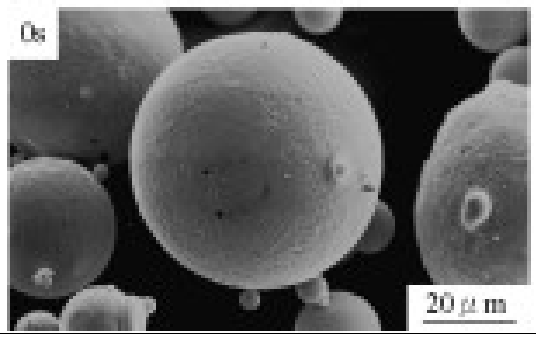


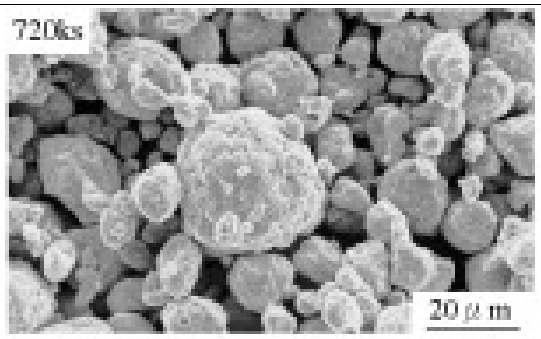
average particle diameter before MM at high kinetic		average particle diameter after MM at high kinetic	
< 900 µm	as received	at 72 ks	< 20 µm

Figure 9: SEM of MM-powder after 0 and 72 ks at high kinetic (CM01)

Figure 10 shows on the left hand side the SEM of the starting powder for the MM at low kinetic which has been sieved with a 200 mesh sieve to < 75 µm for process reasons (see chapter 2.1). The image confirms the same gas atomized powder but in smaller size at an average of about 45 µm. After 720 ks,

which is given in figure 10 on the right hand side, shape also dramatically changed but size not that much. The average size is now estimated to be around 20 µm and the shape is fissured and irregular.





average particle diameter before MM at low kinetic		average particle diameter after MM at low kinetic	
< 75 µm	sieved (200 mesh)	at 720 ks	< 35 µm

Figure 10: SEM of MM-powder after 0 and 720 ks at low kinetic (PBM)

In comparison, in case of MM at high kinetic at 0 and at 72 ks, the average particle diameter was decreased from approximately 120 µm to 20 µm. In case of MM at low kinetic at 0 and at 720 ks, the average particle diameter was decreased from 50 µm to an average particle diameter of approximately 30 µm.

simple comparison of the relations in PS-development, MM-time and absolute powder yield						
MM-process at	starting PS	MM-time	final PS	PS-reduction	comment	powder yield
high kinetic (CM01)	< 900 µm	72 ks	< 20 µm	105 µm	MM-time 10x shorter	200 g
low kinetic (PBM)	< 75 µm	720 ks	< 30 µm	25 µm	PS-reduction 4x smaller	10 g

Table 7: simple comparison of the relations in PS-development, MM-time and absolute powder yield

Table 7 gives some very simple comparison of the relations in PS-development, MM-time and in powder yield. In result, since both equipments used are table-top size, the MM at high kinetic could be considered as far superior here since it gives us 20 times more powder where the particle size is reduced 4 times more in a 10 times shorter time.
Of course this kind of comparison can only give us some limited idea in early stage, however, the difference is that significant, that the preferable route can be determined by this in any case.

3.2 SEM and XRD investigation of MM-powders

We examined the microstructure of the MM powder by SEM and XRD and used the same device as described before.
Figure 11 a-c shows the SEM investigation in cross section of embedded powder samples that has been processed in MM at low kinetic for 0, 180, and 720 ks respectively. The X-ray diffraction pattern given in figure 12 gives us the crystallographic parameters of the same materials where a-e is always marked at each of the single curves.

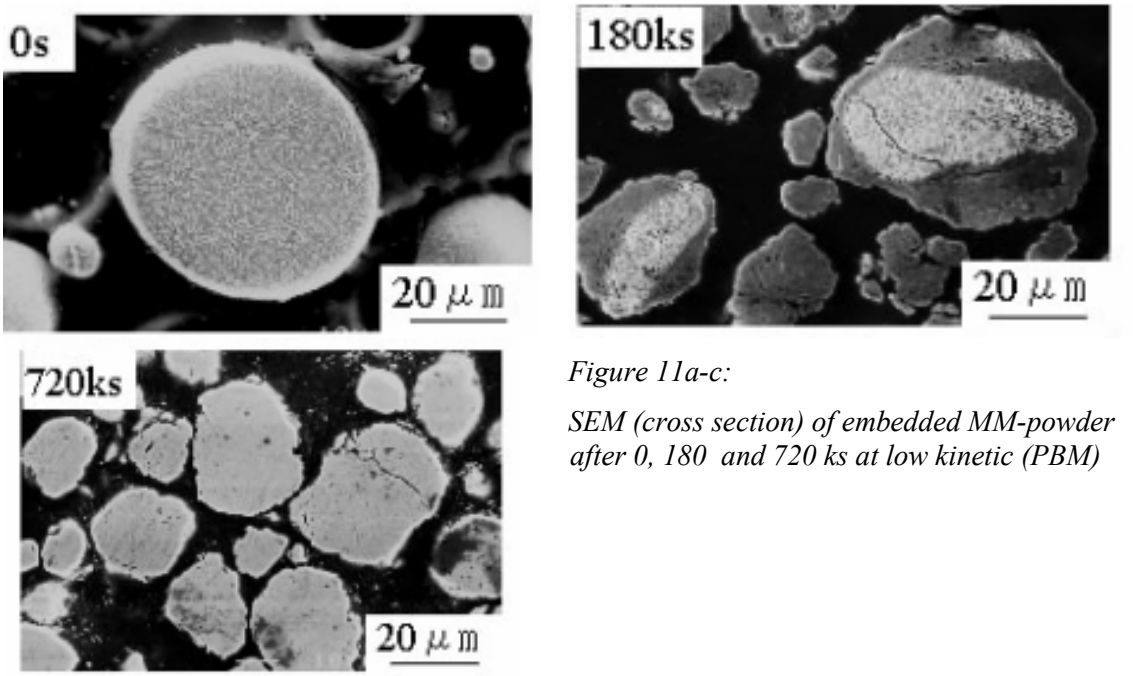


Figure 11a-c:

SEM (cross section) of embedded MM-powder after 0, 180 and 720 ks at low kinetic (PBM)

SEM-investigation confirms the dendritic structure of the starting material where this structure completely disappears after 720 ks MM at low kinetic by grain refinement and destruction which is caused by the deformation effects of the kinetic impact.

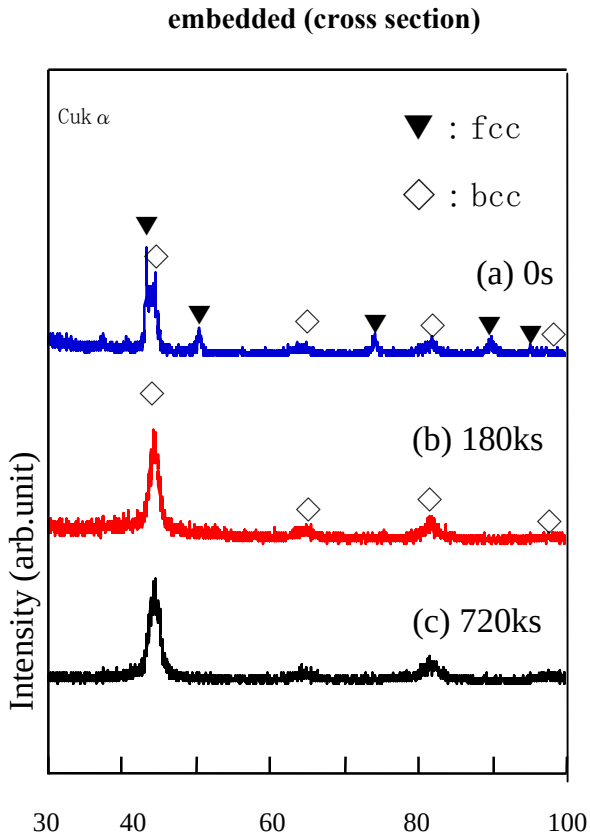


Figure 12: XRD-pattern (cross section) of embedded MM-powder after 0, 180 and 720 ks at low kinetic (PBM)

X-ray diffraction as given in figure 12 confirms, that the inner corpus of the starting powder particles is composed of FCC and BCC structure. The FCC phase is expected to be an austenitic phase that remains from rapid consolidation at gas atomization when the powder was produced. During the MM, the diffraction patterns of FCC disappear and become only the diffraction patterns of BCC. It is thought that the phase of the remained austenite transformed into ferrite (BCC) by strong strain introduced by the MM process. The complete transformation to BCC-phase can be confirmed at 180 ks at low kinetic MM with the PBM which probably happens much earlier but due to the long term operation time experience with the planetary ball mill, the process schedule was set up correspondingly.

Figure 13 a-e shows the SEM investigation in cross section of embedded powder samples that has been processed in MM at high kinetic for 0, 3.6, 18, 36 and 72 ks respectively. The X-ray diffraction pattern given in figure 14 gives us the crystallographic parameters of the same materials where a-e is always marked at each of the single curves.

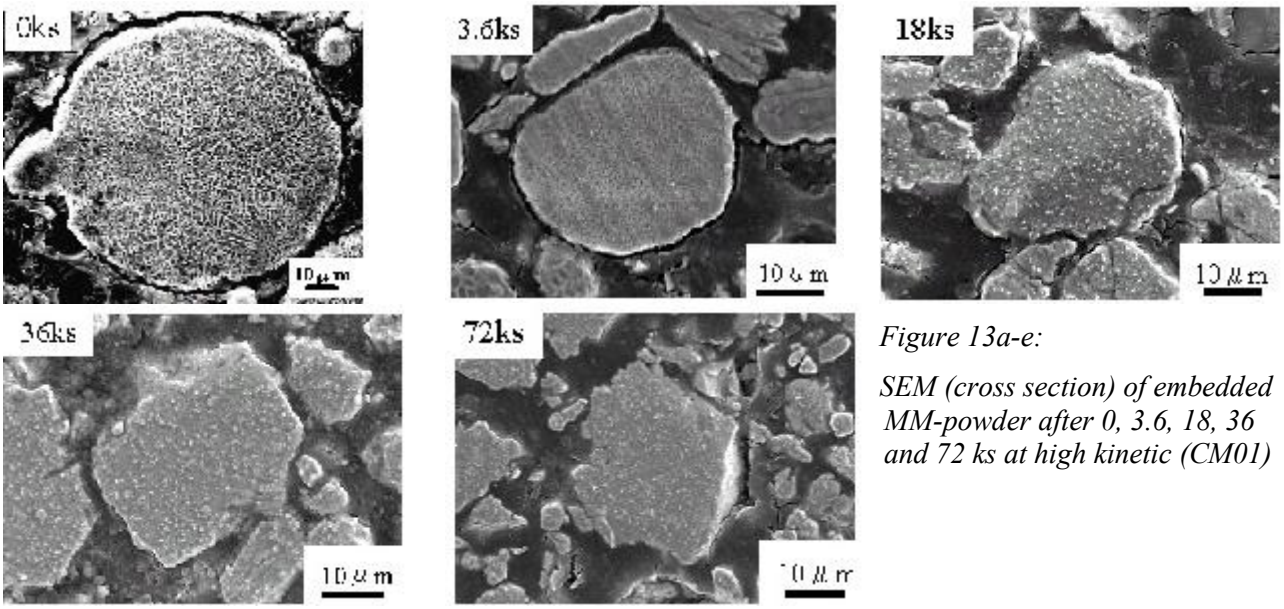


Figure 13a-e:

SEM (cross section) of embedded MM-powder after 0, 3.6, 18, 36 and 72 ks at high kinetic (CM01)

SEM-investigation confirms the dendritic structure of the starting material where this structure completely disappears after 18 ks MM at high kinetic by grain refinement and destruction which is caused by the high deformation effects of the high kinetic impact.
A precipitation in the matrices of the powder can bee found as of 18ks and will be confirmed with XRD (see following) and will be explained in chapter 3.5.

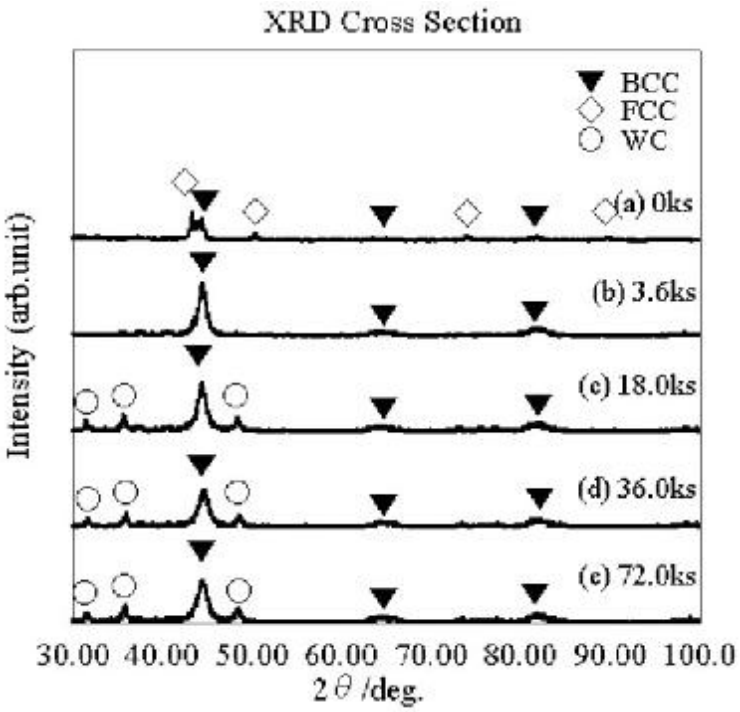


Figure 14: XRD-pattern (cross section) of embedded MM-powder after 0, 3.6, 18, 36 and 72 ks at high kinetic (CM01)

X-ray diffraction as given in figure 14 confirms, that the inner corpus of the starting powder particles is composed of FCC and BCC structure and is completely transformed to BCC-phase at 3.6 ks MM at high kinetic where the same explanation is valid as given in case of the MM at low kinetic, just the transformation-period is tremendously shorter with the CM01. After 18 ks MM we detect the formation of a WC-phase which corresponds to the found precipitations by SEM. This can only be contamination by the rotor of the Simoloyer CM01 where the rotor tips are made by stellite which is a Co-matrix with W- and Cr-carbides. This problematic occurred since unfortunately, a worn off rotor was mixed up to be a new rotor and did cause this severe problematic (see chapter 3.5).

In comparison of the SEM and XRD results of MM-powders at low and high kinetic, the destruction or disappearing of the dendritic structure of the starting material takes place after MM after 720 ks at low kinetic (PBM) and after 2.6 ks at high kinetic (CM01) which would be 200 times faster, however, we are

not sure if this does not take place earlier since the schedule gap between 180 and 720 ks is quite large. So we can state, since it did not happen yet at 180 ks, that the time factor is 50-200. The formation of complete BCC-structure is achieved at low kinetic after 180 ks and at high kinetic after 18 ks which is 10 times faster.

simple comparison of the relations in destruction of dendritic structure, complete formation of BCC-phase and MM-time			
MM-process at	low kinetic (PBM) MM-time	high kinetic (CM01) MM-time	comment
destruction of dendritic structure	180-720 ks	3.6 ks	MM-time 50-200x shorter at high kinetic
complete formation of BCC-phase	180 ks	18 ks	MM-time 10x shorter at high kinetic

Table 8: simple comparison of the relations in destruction of dendritic structure, complete formation of BCC-phase and MM-time

Table 8 gives some very simple comparison of the relations in destruction of dendritic structure, complete formation of BCC-phase and MM-time. In result, since both equipments used are table-top size, the difference as called here low- and high kinetic is confirmed since the kinetic impact is responsible for the deformation of particles and grains and therefore for the destruction of the dendritic structure and introduces high strain into material which is believed to cause the phase transformation as well. Of course this kind of comparison can only give us some limited idea in numbers, however, the difference is that significant, that the preferable route can be determined by this in any case.

3.3 Hardness & microstructure of SPS-samples consolidated from the MM-powders

After the MM-powders were consolidated by SPS, hardness was determined by Vickers test and the microstructure of the MM/SPS compacts was examined.
Figure 15 compares the received hardness-data, where the 5 columns on the left exhibit the date from the samples that have been made from the starting powder (0 ks) and from the powder being MM at high kinetic (CM01) at the different MM-times between 3.6 and 72 ks. The single column to the right gives the data from the sample based on the MM-powder at low kinetic (PBM).

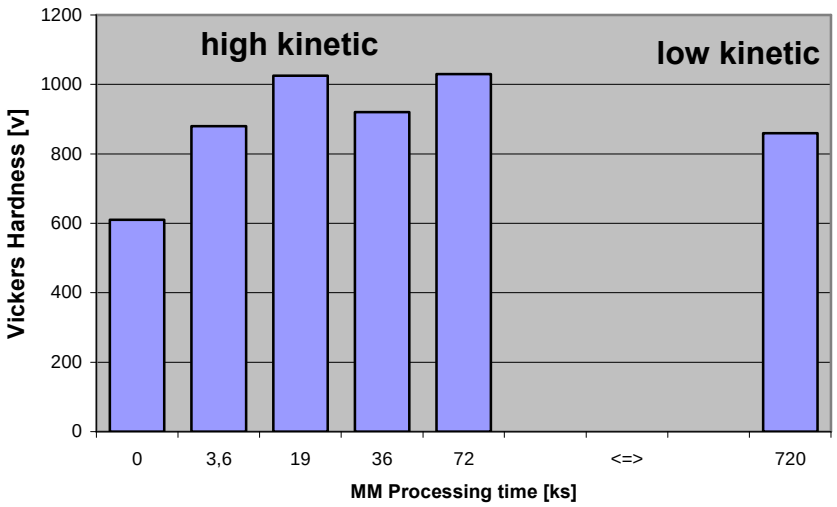


Figure 15: Vickers hardness test of SPS-samples consolidated from the MM-powders

In result, the hardness is increasing with the MM-time for up to 40 % compared to the base material. In comparison, the value achieved after 720 ks MM at low kinetic is slightly exceeded already after 3.6 ks MM at high kinetic. This is in so far important, as at this point we did not see the precipitation nor the WC-phase in SEM/XRD investigation of the as-MM powders yet. Carefully, up to here we can therefore relate it to the more homogeneous and finer distribution of the hard-phases in the HS-steel matrices.

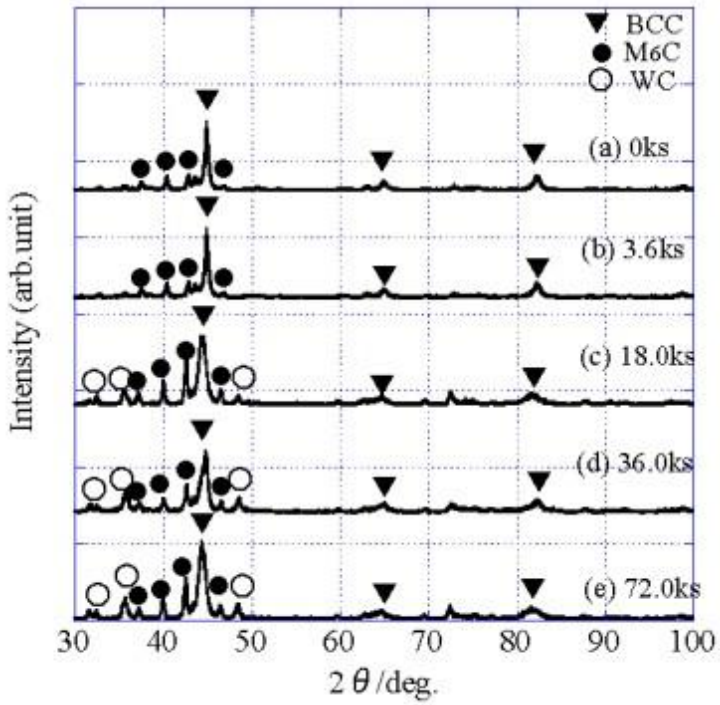


Figure 16: XRD-pattern of SPS-samples consolidated from the MM-powder after 0, 3.6, 16, 36 and 72 ks at high kinetic (CM01)

Figure 16 shows the XRD-patterns received from the consolidated SPS-samples where we this investigation has only been done for the samples related to the starting powder (0ks) and the MM-powder at high kinetic. In result, a very fine grain structure was maintained even after SPS at 1223K for 5 min.

3.4 Comparison on MM/SPS-compacts with conventional HSS by HIP/Forging

Since the SEM-data here is not available yet, we use the results of earlier work [04] to compare the visual microstructure of the MM/SPS (MM and consolidated by SPS) with conventional material produced by HIP/Forging. This is exhibited by Figure 17.

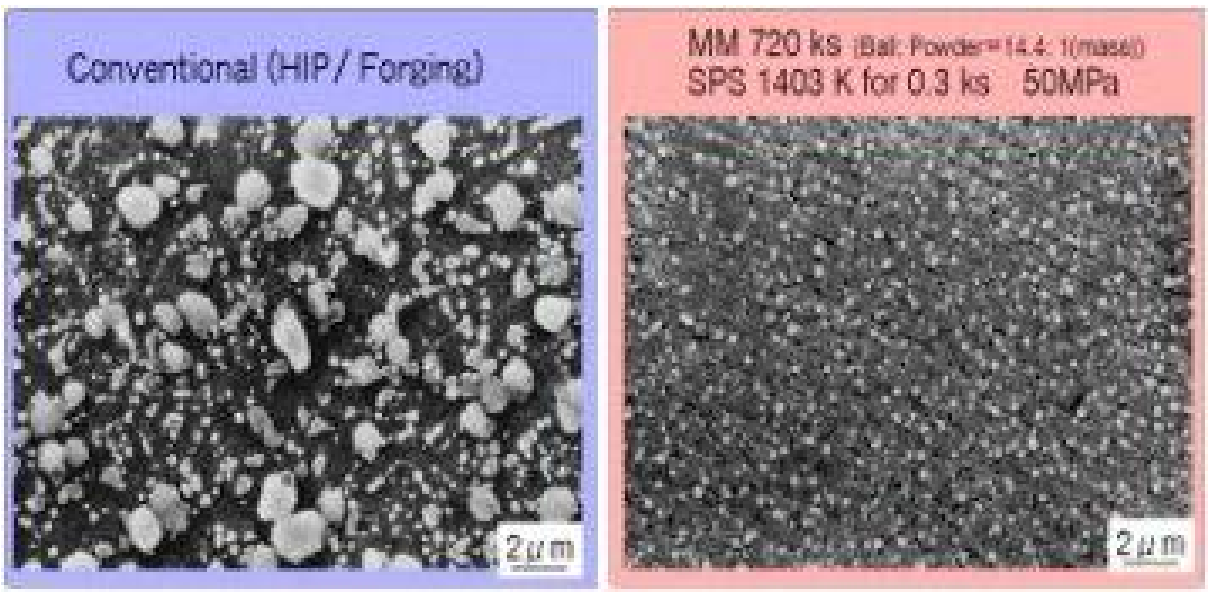


Figure 17a-b: SEM of conventional HSS-compact by HIP/Forging and of MM/SPS compact

The expectation at chapter 3.3 related to the hardness-tests is confirmed since the MM/SPS material shows a much more homogeneous and also a much finer distribution of the hard-phases in the HS-steel

matrices. The average size of carbides and the grain size is expected to be up to 2 micron in case of HIP + forged and less than 0.5 micron in case of MM.

The same materials [2] are compared in compression tests. Figure 18 shows the comparison at 293 K and 823 K test temperature respectively and the results show in both conditions, a superiority of the MM/SPS samples.

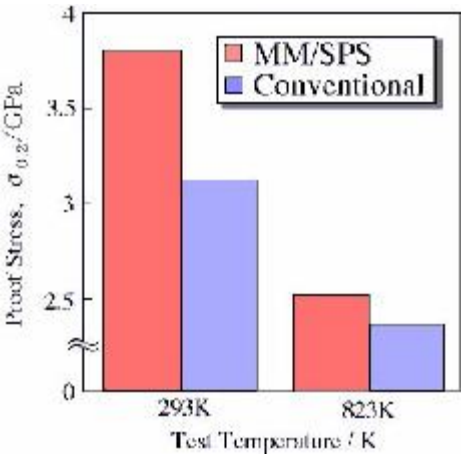


Figure 18: Proof stress of conventional HSS-compact by HIP/Forging and of MM/SPS compact

The 0.2 % proof stress of the MM/SPS compacts were improved by 21 % at room temperature and by 6 % at 823K compared with the conventional compacts.

3.5 Accidental exchange of a worn out milling tool

In this work, accidentally a severe mistake happened in the very beginning and turned out in the very end. Since this kind of waste of resources happens to often at to many sites, this time and here it shall not be just dismissed but discussed.
In mankind, still the human being carries the knowledge by learning and by experience. Even if the simple knowledge data is stored in some technical memory, it is still the human to do the lay out of this. And whenever human beings are moving to wherever, some exchange takes place. And a major issue here must be to not to loose any information for the site at this very moment. In many universities and in many companies and finally in all kind of organizations, every day people are moving to other challenges. And this is necessary and favorable for many good reasons. However, we must very carefully take care that any basic and important information is transferred before the exchange.
What happened here is, that a high-tech milling device had been operated by some students at the university and before the next projects were started with new young scientists, the device was neither operated nor the information how to use it in detail was transferred. Just a few weeks were enough to loose significant information for the site, in this case the laboratory. So finally the newcomers found a fully worn out rotor which means the entire device was not in usable condition at all. However, as of a lack of information, it was believed it would be.
That means here, the MM at high kinetic of the HS-steel powder was done with a rotor that could not do it at all and in fact it is surprising how good the results still were. Actually most of the targets as far as they are related to the comparison attempt of high and low kinetic could still successfully be proved.

new usable rotor
this is how it should be

already worn out rotor
this was falsely taken as a new rotor !

completely destroyed rotor after long term operation at no usable condition

Figure 19: condition of the rotor of the CM01

Figure 19 shows on the left hand side the rotor of the Simoloyer CM01 in new condition. The picture in the middle shows the rotor in fully worn out condition which means it should have been replaced right away. But exactly this condition was accidentally believed to be a proper condition for operation and of course after relatively short time of maybe 60 h, this rotor change to the condition as shown on the right hand side in figure 18. And what then happened is of course, that the remaining carbides on the worn out rotor tips were very fast distributed in the product-matrix since they were kind of eroded out of the austenitic matrix of the rotor base. And this is why we find the precipitates in the corresponding SEM-investigation and this is why we find the WC-phase in X-ray diffraction investigation. Certainly the material of the rotor base blades also contaminated heavily the product but could not be noticed since it consist of the same elements than present in the HS-steel material.

4 Conclusions and References

4.1 Conclusions

High speed steel powder has been processed by Mechanical Milling and consolidated by Spark Plasma Sintering in order to improve microstructure and mechanical properties.
Mechanical Milling has alternatively been performed in a planetary ballmill Fritsch P-5 and in a horizontal high energy ballmill Simoloyer CM01. Both devices are considered as table top equipment where the Simoloyer has been found to be far superior in terms of processing time, powder yield, absolute processing capability, handling and temperature control.
In terms of particle size reduction versus processing time, the Simoloyer is 10 times faster and produces 20 times more powder at also much higher size reduction value.
The powder yield in the Simoloyer has been found to be nearly 100 % where the data for the planetary ballmill proves a yield below 50 %.
The particle shape in the Simoloyer gets more flattened due to tremendously higher kinetic impact which is also proved by the facts that the dendritic structure of the HSS starting powder is disappeared 50-200 times faster and the transformation to complete BCC-structure is noticed 10 times faster when using this device instead of the planetary ballmill.

Spark Plasma Sintering has been successfully used to consolidate MM-HS-steel powder samples under maintaining a very fine microstructure of the materials.
Compared to conventional material, the MM/SPS-material exhibits more homogeneous distribution of the carbides at a much finer and smaller structure. The MM/SPS-material is superior in hardness and proof stress.

Fortunately, most of the superiority proving results with the Simoloyer were achieved already at MM-times below 18 ks because as of this processing time, significant contamination (WC) by the rotor was found which has been caused my a mistakenly chosen worn out rotor where it has not been noticed that this rotor would have had to be restored.
In the future, the material shall again be processed with the Simoloyer but then in good tool-condition where even a higher kinetic impact and no severe contamination must be expected. Then the SPS-compacts of this material shall be compared with the conventional HSS-material.

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