Paper will be published at the International Thermal Spray Conference, ITSC 2002, Essen, Germany, 4 th – 6 th March 2002

Fast Quality Control of Spray Powders
Schnelle Pulverkorngrößenbestimmung zur Qualitätskontrolle

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Die Qualität thermisch gespritzter Schichten wird entscheidend durch die Qualität der verwendeten Spritzzusätze bestimmt. Neben der chemischen Zusammensetzung sind bei pulverförmigen Spritzzusätzen die Fliessfähigkeit, die Schüttdichte und insbesondere die Pulverkorngrößenverteilung von großer Bedeutung. Lassen sich die Fliessfähigkeit und die Schüttdichte durch relativ einfache Prüfmittel näherungsweise ermitteln, so ist die Bestimmung der Pulverkorngrößenverteilung mit erheblichem zeitlichen und apparativen Aufwand verbunden. Spritztechnische Betriebe müssen daher in der Regel auf die Angaben der Pulverhersteller zurückgreifen oder sich diese Daten von einer externen Prüfstelle im Auftrag messen lassen. Eine Prüfung Vorort, idealerweise direkt vor der Verwendung des gewünschten Spritzzusatzes ist nicht oder nur eingeschränkt möglich. Ein neues, einfaches und preisgünstiges Prüfverfahren auf der Basis vollautomatisierter Bildanalyse in Kombination mit einem konventionellen, hochauflösenden Diascanner anstelle eines Durchlicht- oder Elektronenmikroskops zur Objekterfassung, bietet hier eine interessante Alternative. Dieses Verfahren ist einfach zu bedienen und liefert innert weniger Minuten Informationen zur Korngrößenverteilung und zur Kornform. Ziel dieser Arbeiten ist der Vergleich dieser neuen Prüftechnik mit im spritztechnischen Alltag etablierten und standarisierten Prüfmethoden wie der Siebanalyse nach ISO 4497 oder der Pulverkorngrößenbestimmung mittels Laserstreulichtverfahren.

The quality of thermally sprayed coatings is strongly influenced by the quality of the spray powder. Most important powder properties are: chemical composition, flowing behaviour, apparent density and particle size distribution. Flowing behaviour and apparent density can be checked relatively easy. In opposite, the determination of the chemical composition and of the grain size distribution is time intensive and complex. Therefore, often times the end-user has to trust the information given by the supplier or he gives the analysis to an independent institute. For quality control it would be ideal to measure the grain distribution before spraying. A new, simple and low cost measuring technique offers a new alternative. This system combines a modern slide scanner with a fully automatic image analysis system specialised on powder analysis that is fast and easy to use. Aim of this work is to present this new technique in comparison to powder analysis methods that are established in the thermal spraying society and / or standardised, as sieving analysis and laser scattering methods.

1 Introduction

Metallurgical powders can be characterised by different techniques as there are image analysis, size analysis using viscous fluid, laser particle size analysis, sieve analysis or particle surface area measurements by using adsorption of gas on the surface (BET) or using the gas permeability (Fischer Sub Sieve Size). In thermal spraying the standardised sieving analysis according ISO 4497 and laser particle analysis methods are established.

Sieving analysis is relatively time consuming and requires a complex set of sieves different in mesh size to achieve useful results. If the steps between the sieve classes are too large the information on particle size distribution for thermal spray powders are limited or even useless. Another problem is the limitation to minimum mesh sizes below 20 µm. Although, finer powder cuts down to a minimum particle size of 5 µm are of increasing importance in the thermal spray business. In case of coarser powder cuts the determination of the so-called dust contents is very critical. For example, to high dust content of a powder can causes plugging of the nozzle of a spraying torch or may increase the oxide contents of metallic layers sprayed with this powder. Laser particle sizers commonly determine the dust content of powders. This type of equipment is very expensive and the service needs a specialist. Another problem, there a different laser particle analysers on the market and sometimes the

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powder specification requires the test by a laser particle analyser of the supplier x and in the other case of the supplier y. Therefore, conventional spray shops are often not equipped with laser particle analysers.
A new particle analyser called “Powdershape” could be a low cost, easy to apply and reliable solution of the above mentioned problems. The Powdershape system is a combination of a high resolution slide scanner (4000 dpi) and a specialised, fully automatic image analysis system. This new measurement device allows not only the fast determination of the particle size distribution as sieving analysis can do it. It delivers also results on dust contents or additional information on powder shape. Latter one couldn’t be even measured by laser particle analysers. Although, the question rises up, how far results achieved by the “Powdershape” are comparable to the established particle analysis methods and what is the theoretical concept behind.

2 Concept of „Powdershape“

2.1 Why using a scanner instead of microscope

Since few years scanners are on the market with pixel sizes down to 5 µm. 4000 dpi slide scanners have typically pixel sizes down to 6 µm. This pixel size leads to images comparable with microscope magnifications up to 35 x. Slide scanners work therefore in the same range of resolution, which is necessary for powder inspection.
A scanner has in contrast to classical microscopes with video cameras four major advantages:

1. The observed field with up to 4000 x 5888 pixel is much larger than the field observed with a camera mounted on a light microscope (image size typically 512 x 768 pixel)
2. The illumination of all image points is the same, the focus is done automatically. This fact enables to make highly reproducible images. The reproducibility is a very important condition for any kind of quantitative image analysis.
3. The scanner became much more cheaper than a microscope - video camera solution during the last few years.
4. The combination of good optical resolution with large CCD-arrays enables measurements of thousands of powder particles at the same time. Thus, a statistical evaluation becomes very easy.
5. Based on these obvious advantages of slide scanners the “Powdershape” system has been developed especially for quality control of metallurgical powders.

2.2 Measurement parameters

2.2.1 Particle size

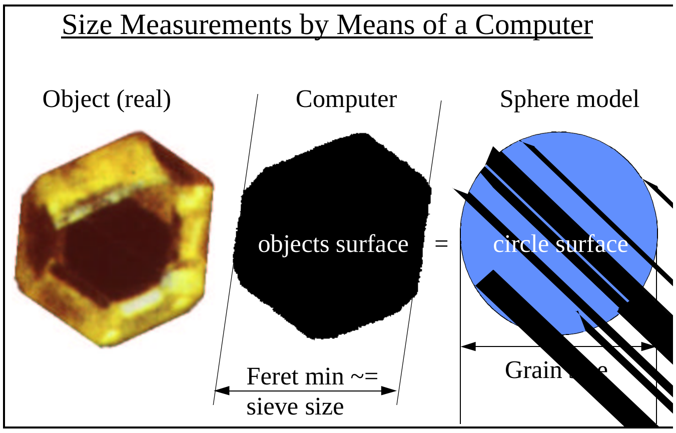

Fig. 1: **Definition of size measurements**
Bild 1: **Definition der Partikelgrössenmessung**

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The **grain size** used by particle sizers is in general defined as the diameter of a sphere having physical properties that are most similar to the measured properties of the real particle. The grain size is easy to calculate in the case of image analysis just by measuring the surface area and evaluating the diameter of a surface equivalent circle, fig. 1.
The **sieve size** is not directly available because the image is only a two dimensional projection of a three dimensional object. In the Powdershape the minimum diameter Ferret min and the diameter of the minor axis of an ellipse with the same geometrical momentum of inertia and surface area are used. Therefore these calculated sieve sizes may be used for a comparison with real sieve size measurements, but the given values are just an approximation and limited to different three dimensional constructed idealized shapes such as regular polygons and ellipsoids respectively.

2.2.2 Particle shape

Three well-known shape factors exist in correlation to a circle using the outline of an object:

- The **elliptical shape factor** f2 e.g. major axis/ minor axis an ellipse same geometrical momentum of inertia. This factor is very useful for description of unidirectional elongated diamonds but not suitable for the different state of cristallinity because it cannot distinguish between circular symmetric polygons as squares and hexagons.
- The **Ferret-ratio** that is the ratio between the minimum diameter of the object divided by the maximum diameter of the object. This ratio is 1 if the object is a circle and should therefore be a good shape factor for the correlation of an object to a surface equivalent circle. However this factor only works well if the outline line is everywhere convex and leads to wrong interpretations if the measured particle shows concave outline parts.
- The **circular shape factor** f. f is calculated from the surface equivalent diameter of a circle Uc and the real particle perimeter shape:

$$f = U/Uc(F)$$

U... particle perimeter, Uc(F) perimeter of surface equivalent circle, F ... particle surface.

The factor f is the root of the size independent shape factor S that is well introduced in the field of digital image processing; see F. M. Wahl [1] and E. Exner [2]:

$$S = U^2/(4\pi F)$$

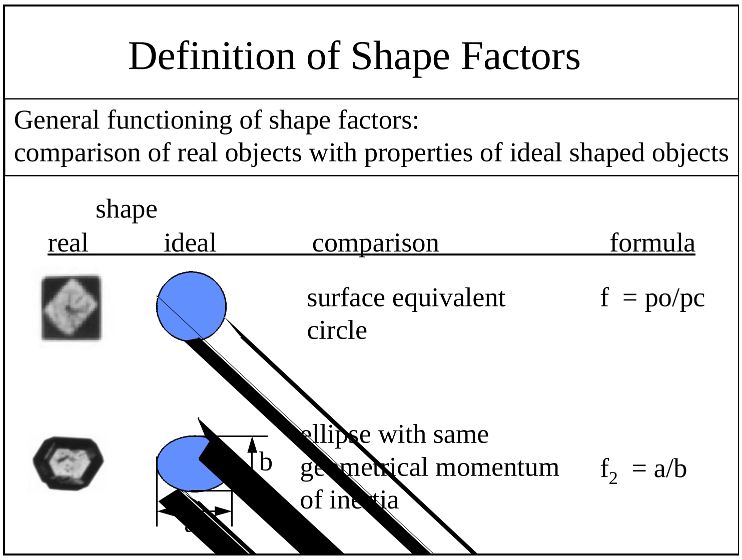

Fig. 2: Definition of shape factors: circular shape factor f, elliptical shape factor f2
Bild 2: Desfinition des Formfaktors: Kreisformfaktor f, elliptischer Formfaktor f2

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For the characterization of thermal spray powders the elliptical shape factor was determined as the numerically most stable one.
The “Powdershape” is based on a software developed for quality analysis of diamonds, called DIASHAPE. Therefore, more details on the theory behind and way of system calibration are still published by H.G. Schmid et al. [3, 4].

3 Experiments

For comparison of the results achieved by different particle analysing techniques powder specimen were taken from 4 WC-Co powders different in particle size range (5 – 25 µm, 20 – 45 µm, 5 – 45 µm, 45 – 106 µm). Sampling was done according ISO 3954.
Sieve analysis of this powder samples were done according ISO 4497 and the “dust contents” was measured using a laser particle analyser of Microtrek. One powder lot was also characterised with a laser particle analyser of Malvern. In case of particle analysis with the “Powdershape” a small quantity of the powder to be analysed has to be placed on a very clear Scotch tape fixed in a slide frame. After scanning this slide frame with powder the software is running fully automatically, saying no illumination levels, filters a.s.o. has to be fixed by the operator. On a single slide the system will identify normally several thousand particles of a thermal spray powder. Here minimum 5000 particles per slide were measured. Measurement time for one powder lot was less than 5 minutes, using a 1.3 GHz PC.

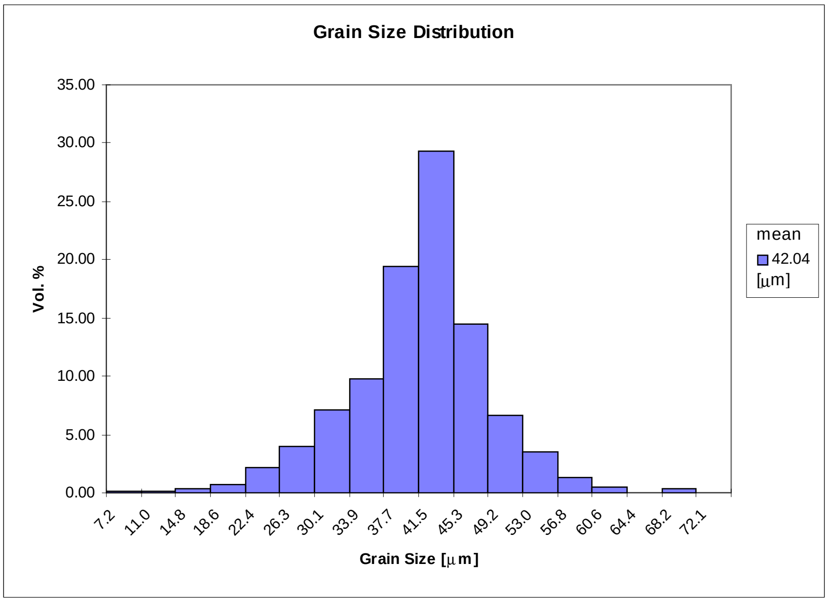

Fig. 3: Particle size distribution of 20 – 45 µm WC-Co powder measured with Powdershape
Bild 3: Pulverkorngrössenverteilung eines 20 – 45 µm WC-Co-Pulvers, gemessen mit Powdershape

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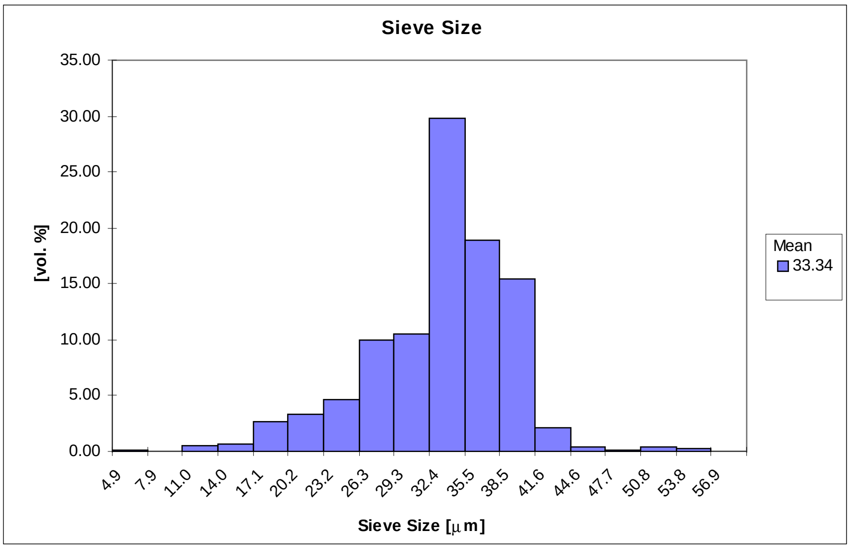

Fig. 4: Sieve size distribution of 20 – 45 µm WC-Co powder measured with Powdershape
Bild 4: Siebanalyse eines 20 – 45 µm WC-Co-Pulvers, gemessen mit Powdershape

4 Results and discussion

Typical print outs of particle size measurements with the Powdershape are shown in Figure 3 and 4. The results of the sieve size analysis tending to lower values due to physics.
The shape factor of the same powder as function of the particle size is given in figure 5. The sieving edges at 20 µm and 45 µm are clearly visible as large steps in the shape factor of the powder.

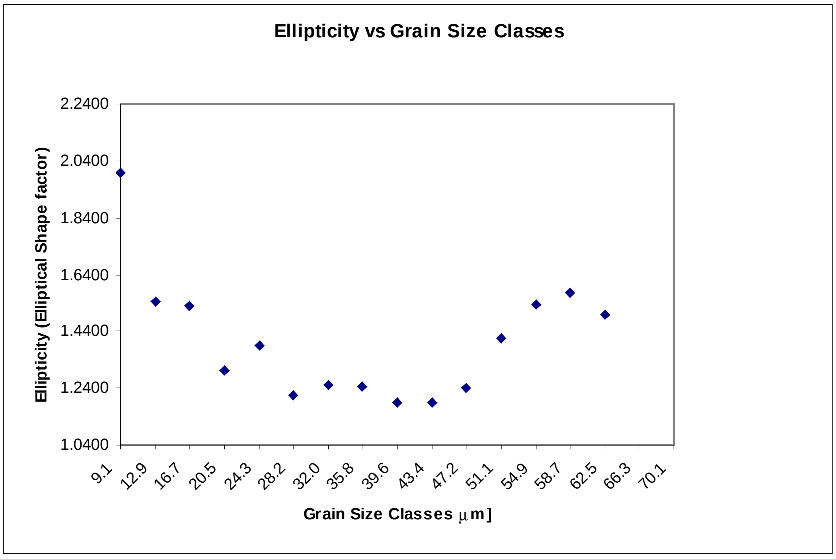

Fig. 5: Ellipticity versus particle size of a 20 – 45 µm WC-Co powder determined with Powdershape
Bild 5: Elliptischer Formfaktor versus Pulverkorngrösse eines 20 – 45 µm WC-Co-Pulvers, gemessen mit Powdershape

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Table 1 gives the average grain size and standard deviation of the different powders measured by the different techniques.

	Sieving Analysis		Microtrek		POWDERSHAPE							
					by volume		by number		sieve model			
Grain size range in µm	Mean µm	S.D. µm	< X µm	%	Mean µm	S.D. µm	Mean µm	S.D. µm	Mean µm	S.D. µm	< X µm	%
5 - 25	16.4	9.1	5	2.16	12.9	4.1	9.3	2.9	9.2	2.5	5.8	2.16
20 - 45	32.2	5.4			42.0	15.1	33.4	12.0	33.3	12.4		
5 - 45	27.1	11.5	5	0.15	30.5	16.8	16.1	8.8	23.5	13.4	5.2	0.15
45 - 106	71.4	18.4			76.6	17.0	66.6	15.1	70.1	16.3		

Tab. 1: Measurement results of powder analysis by different techniques
Tab. 1: Messresultate verschiedener Prüftechniken zur Pulverkorngrössenanalyse

The values of the sieve model used in the "Powdershape" are very close the values by classical sieving analysis if the average particle size is above 20 µm. Although, the classical sieving analysis doesn't allow a real measurement of the particle size below 20 µm, it just gives the information that a percentage of the powder is below 20 µm. A better image how far the results are comparable allows figure 6. Here all measurement results of the sieving analysis of a single powder lot are in direct comparison to the results get by the sieving model of the Powdershape for the same powder lot and the same steps in mesh size ranges.
Figure 6 demonstrates that the measurement results for both measurement techniques are nearby identical.

The dust content of the powder (here defined as quantity of powder particles below 5 µm) is determined by Microtrek in case of the powder 5 – 25 µm with 2.16 % and for the powder 5 – 45 µm with 0.15 %. This percentage values were given into the image analysis software that finally calculates the maximum particle size determined for the same powder lots at this percentile. It is 5.8 µm in case of the powder 5 – 25 µm and 5.2 µm in case of the powder 5 – 45 µm. These results are indicating that laser particle analysis with a Microtrek system and the Powdershape delivering more than less identical results for the determination of the dust contents.

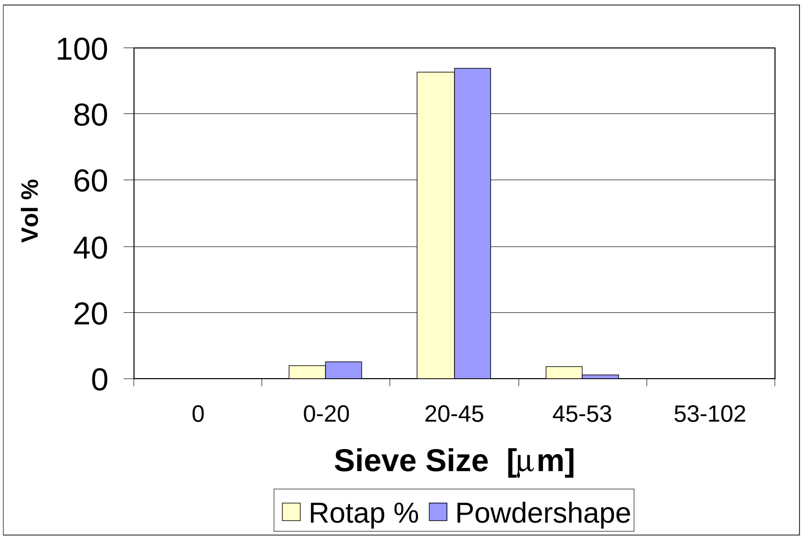

Fig. 6: Results of sieve analysis and calculated sieve model of image analysis of a 20 – 45 µm WC-Co powder
Bild 6: Siebanalyse mittels Sieb und Powdershape im Vergleich, Pulver: 20 – 45 µm WC-Co

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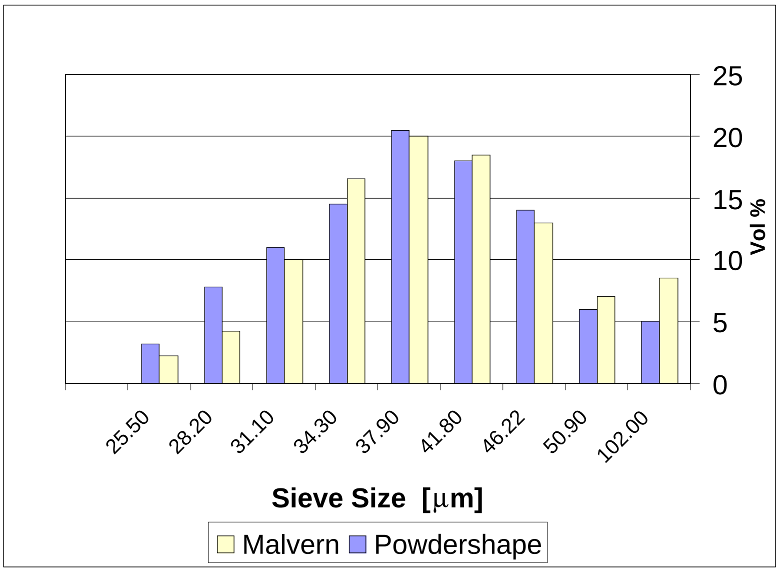

Fig. 7: Particle size distribution of 20 – 45 µm WC-Co powder measured by laser scattering and by image analysis (calculated sieve size)
Bild 7: Pulverkorngössenanalyse eines 20 – 45 µm WC-Co Pulvers mittels Laserstreulichtverfahren und mittels Bildanalyse

That laser particle analysers and the Powdershape delivering comparable results is also demonstrate by figure 7. Here the results achieved by laser particle analysis using a Malvern system are in direct comparison to the results achieved by the Powdershape for the same powder. Both histograms are again very close.
To prove the reproducibility of the results produced by the Powdershape system for each of the 4 different powders described in table 1 at minimum 5 independent measurements were done by three different operators taken all time a new powder sample. The following figures 8 and 9 showing the mean value of particle size, standard deviation and mean shape factor as function of the different powder lots and measurements.

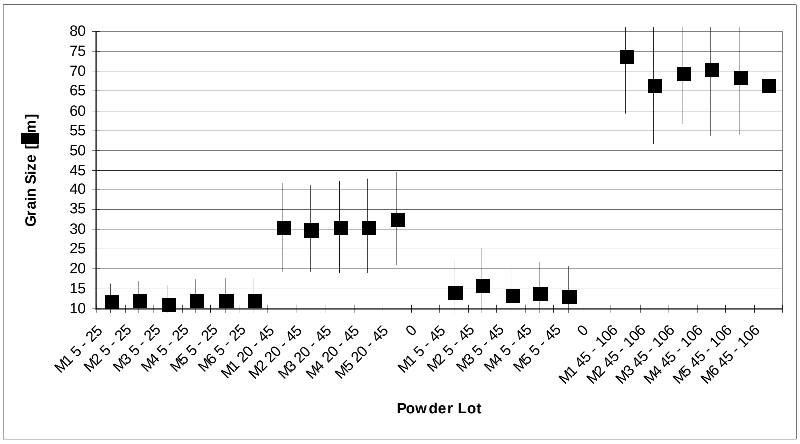

Fig. 8: Mean values and standard deviation of average particle size (number percent) of 4 different powders and different measurements
Bild 8: Mittelwerte und Standarabweichung der Pulverkorngrösse (bezogen auf Anzahl gezählter Partikel) von 4 verschiedenen Pulvern und unterschiedlichen Messreihen

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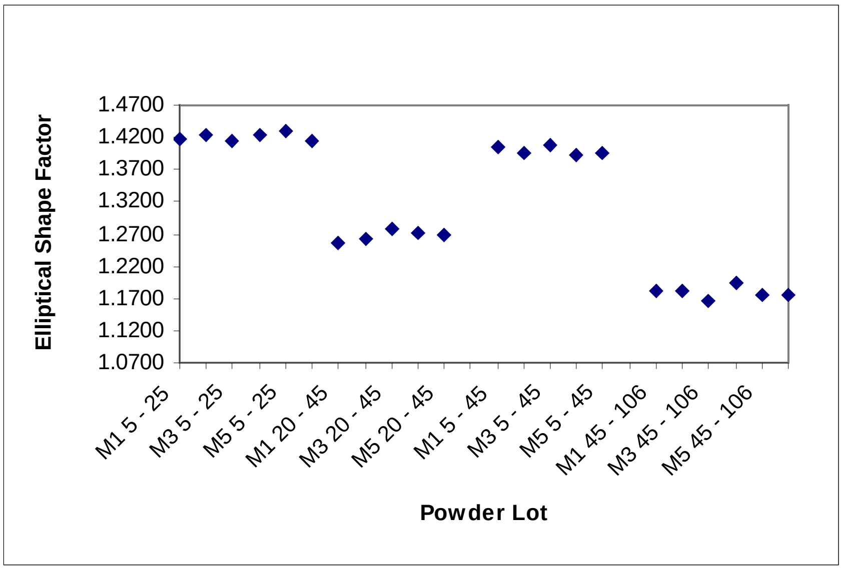

Fig. 9: Mean values of average shape factor of 4 different powders and different measurements
Bild 9: Mittelwerte des elliptischen Formfaktors von 4 verschiedenen Pulvern und unterschiedlichen Messreihen

This two final graphs demonstrating that each powder analysis delivers for each investigated powder a consistent and reproducible result. Sampling causing no remarkable effect on the final result.

5 Summary and Conclusions

A new, fully automatic image analysis system specialised on powder analysis in combination with a high resolution slide scanner allows the fast and reproducible determination of particle size distribution and the shape of the powders. The results achieved with this system are comparable to results of powder analysis by laser scattering methods and by classical sieving analysis.
The additional information on the shape of the powder visualizes sieving edges and is furthermore a sensitive indicator of changes in powder quality.

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