

# Comparison of different measuring methods for the determination of the particle size of powders for plasma spraying

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*The average grain size, the grain-size distribution, the morphology and the microstructure are fundamental characteristics of a spraying powder. Now that the significance of the grain size for the pattern of properties of the powder has been recognised, greater consideration is also being given to it in standards and regulations. However, unfortunately, the processes according to which the grain size and the grain-size distribution must be determined are specified in the rarest of cases. The contribution therefore dealt with the comparison of different particle-size measuring techniques, such as diffraction spectroscopy, sedimentation, sieving and microscopic measurement. The comparability of the measured results was investigated on twelve plasma-spraying powders with different compositions, nominal sizes and morphologies.*

## 1 Introduction and objective

In the chemical industry, in power stations and in a large number of other branches of industry, demands which, in many cases, can only be fulfilled by applying surface coats are being made with regard to the surfaces of certain components. One customary process for the application of such coats is plasma spraying which is used for the coating of components, for example, in order to increase the wear resistance (at high temperatures as well), to guarantee the corrosion resistance or to achieve thermal insulation. A constricted arc which emerges from the torch with high kinetic and thermal energy and entrains the radially fed consumable in powder form is used for this process. The powder particles are, at least in part, in a fluidised state when they strike the workpiece surface with high kinetic energy. In this case, only partial welding occurs. The adhesion is mainly based on adhesive bonding forces and on mechanical interlocking.

The physical and mechanical properties of a sprayed coat are fundamentally dependent, amongst other items, on the quality of the spraying powder. The chemical composition, particle-size distribution and flowing behaviour of the powder are of particular interest. It is primarily the average diameter of the sprayed particles which is of decisive significance for the flawless quality of a sprayed coat. For example, a coarse-grained powder requires a greater quantity of heat for complete fusion and melting-on. On the other hand, the adhesion is more problematical in the case of a finer powder because of the lower kinetic energy when it strikes the base material. Furthermore, the diameter of the sprayed particles is important because, as the grain size

increases, the porosity of the sprayed coat rises and its thermal conductivity decreases [1]. The grain-size distribution plays a decisive role not only in the case of atomised and molten powders. The morphology and composition of the initial powders are extremely important for the properties in the case of micropellets as well.

After the influence of the grain size was recognised not such a long time ago, this is also being increasingly included in national and international regulations and standards as a parameter. Unfortunately, the processes with which the grain size must be determined are rarely indicated there. Time and again, this results in differences of opinion between powder manufacturers and users. The objective of this contribution is therefore to compare and assess measured results from powder grain-size analyses which were carried out according to different measuring processes.

## 2 General discussions about grain-size measurement

In order to be able to estimate the influences of the powder quality and in order to characterise powders, their particle-size distribution, amongst other items, is measured. In this respect, statistical processes serve to investigate a sample and to determine the particle collective. The analysis must be made from a random sample consisting of several individual samples so that the particle-size distribution determined is also representative of the entire powder with an adequate reliability of the statement. The particle size is defined unambiguously by the diameter only in the case of a spherical shape. On the other hand, for forms deviating from the spherical shape, measurable physical properties, such as the projection surface, the settling speed or the diffraction phenomena, are used in order to conclude the diameter of an equivalent sphere on the basis of these properties. In most cases, the particles are separated into fractions with certain diameters and the relative proportions of the fractions are determined in relation to the entire analysis sample. Usually, the relative weight of the individual fractions is related to the total weight. For purposes of graphic representation, the weight totals are calculated by cumulating the percentage weight proportions and are applied above the particle diameters. The grain-size characteristic curves drawn up in this way show passage totals indicating which percentage of the powder weight is smaller than an interesting particle size.

There are already a large number of publications dealing with the determination of the particle size and with the processes in order to describe the particle-size distribution [2...9]. However, articles investigating the comparability of the results determined according to different processes and using different devices are considerably rarer. If any such comparisons do exist, then consideration was only given to the results of different analysis devices in the case of a certain powder. On the other hand, the work dealt with here

compares not only different methods and devices but also their behaviour in the case of very different spraying powders.

### 3 Description of the powders selected

Twelve plasma-spraying powders with different chemical compositions, morphologies and nominal grain sizes were selected in order to compare the different analysis methods for the particle-size distribution. The precise data of the powders is listed in Table 1.

For the investigation, the powders were divided into three size classes and were given corresponding designations. The letter C (coarse) in the powder designation identifies the coarser powders. The M (medium) stands for an average grain size and the F (fine) powders are the finer types. Moreover, the three size classes are each composed of four individual powders which also have different morphologies as a result of different manufacturing processes.

### 4 Analysis devices used

Individual analysis devices with different measuring principles were selected from the large number of possible analysis devices. The manufacturers and the company designations of the devices are not named during the subsequent interpretation of the measured results. The following analysis devices were used:

- Diffraction Spectrometer 1,
- Diffraction Spectrometer 2,
- Sedigraph 1,
- Sedigraph 2,
- Sieve,
- Ultrasonic sieve,
- Microscope.

### 5 Test results

For the graphic representation of the particle-size distribution, the percentage passages of the seven analysis devices were applied above the equivalent particle sizes. For the twelve powders, this results in the cumulative passage distributions shown on Figs. 1 to 4. However, since seven characteristic curves in one diagram make this fairly unclear, a shaded area where the majority of the measured results are located is marked on every figure. This area does not include those results which were assumed to be subject to systematic deviations, for example by sampling, or individual measuring errors.

## 6 Comparison of the results

In accordance with literature results, the comparison of the results on Figs. 1 to 4 shows that a specific statement is only permissible with regard to one special powder. The consideration of two powders with the same chemical composition but with a different nominal grain size (for example, the 3M and 3F powders containing chromium oxide) thus results, on the one hand, in a usable comparison of the particle-measuring techniques but, on the other hand, in extreme deviations in the cumulative passage distributions. Therefore, it must not be concluded from the analysis of the 3M powder, in which the distributions were relatively close together, that the analysis of the 3F powder also provides comparable values. However, it is noteworthy that the measured results for the powders with medium and fine grain sizes tend to be more similar than in the case of the coarse C powders.

When the results of the diffraction spectrometers are compared, it must be noted that these differ with regard not only to the software and error correction but also to their calibration. Therefore, Diffraction Spectrometer 1 generally determined a lower passage, thus portraying the investigated powders as being coarser. Especially as from a particle size of approximately 40  $\mu\text{m}$ , there is extreme divergence of the measuring curves. However, the lower the coarse proportion of a powder is, the more similar are the results. In the case of the fine powders, it was, in part, even possible to establish correspondence. On the other hand, the comparison is problematical in the case of the coarse powders because Diffraction Spectrometer 1 assumes a totally different distribution here. The diameter definition of the process cannot be accused of this. Here, it is presumably other influences, such as the type of evaluation by the software, which take effect.

On the other hand, the measured results of the two devices which make use of the sedimentation in order to analyse the particle size are satisfactory. The two sedigraphs which had the same construction type but were operated in different laboratories with, in part, different liquids for the suspension provided reproducible results. It was shown that there was good correspondence between the cumulative passage distributions determined. Although minor deviations occurred, these may be explained by the sampling, by minor deviations of the measuring temperature and by the agglomeration behaviour of the particles.

From the representation of the measured results on the figures, it is also possible to recognise the comparability of

Table 1. The plasma spraying powders analysed.

powder	manufacturer's designation	composition	nominal size $\mu\text{m}$	manufacturing type	morphology
1C	AMDRY 3269	Cu	-90 +45	gas-atomised	spherical
1M	AMDRY 4535	Ni20Cr	-53 +16	gas-atomised	spherical
1F	AMDRY 4532	Ni20Cr	-30 +5	gas-atomised	spherical
2C	-	Mo	-90 +45	agglomerated and plasma-compressed	spherical
2M	AMDRY 983	WC17Co	-63	agglomerated and plasma-compressed	spherical
2F	-	ZrO <sub>2</sub> 2MgO	-25	agglomerated and plasma-compressed	spherical
3C	AMDRY 6208	Al <sub>2</sub> O <sub>3</sub> 3% TiO <sub>2</sub>	-90 +45	molten and broken	blocky/angular
3M	AMDRY 6410	Cr <sub>2</sub> O <sub>3</sub> 99.7%	-45 +22	molten and broken	blocky/angular
3F	AMDRY 6400	Cr <sub>2</sub> O <sub>3</sub> 99.7%	-22 +5	molten and broken	blocky/angular
4C	AMDRY 3039	Al 99%	-106 +30	water-atomised	spattered
4M	AMDRY 3033	Al 99%	-45 +5	water-atomised	spattered
4F	-	Al 99%	-30	water-atomised	spattered

the sieve analyses with each other. The reasons for the minor deviations can be found in the sieving duration and in the sieving quantity. These were stipulated according to empiric values. Unfortunately, the results from the dry and ultrasonic sieve analyses can only be compared in the case of the coarser powders because the measuring ranges hardly coincide otherwise. In general, the sieve analyses must be assessed critically in the case of the fine powders since the tolerances with the nominal-width openings of 5 and 10  $\mu\text{m}$  have a major influence on the passage and may distort the results. In comparison with other measuring processes, the passage through these sieve foils was too great.

A comparison of the sedimentation results with those from the sieve analysis which, because of the connections described in the previous paragraph, were mainly determined using the ultrasonic sieve shows the trend that the sedigraphs portray the powders as being finer. In the medium grain-size range, the average difference is approximately 10% of the passage. However, it was shown that overlapping was possible in most cases by means of parallel displacement of the curves. It may be concluded from this that an identical distribution was determined, but with another particle size. The difference between the two processes could therefore be caused by another physical diameter definition.

In the case of the assessment of the results of the sedigraphs in relation to those of Diffraction Spectrometer 2, which calculated similar distributions, consideration must be given to the influence of measures which were taken for dis-agglomeration purposes. Thus, sodium pyrophosphate was added to the suspension during the sedimentation process as a deflocculant. As a result of this, the sedigraphs portrayed the corresponding powders as being finer.

The sedigraphs and the diffraction spectrometers also measured the hollow and porous powders as being finer. Here, the influence of the porosity on the diffraction and the non-homogeneous density became apparent during the sedimentation process. There was astonishingly good correspondence between the devices in the case of the analysis of spattered particles in so far as the powder did not predominantly consist of absolutely coarse particles which, due to their position, can exert a major influence on the measurement.

The best correspondence of the cumulative weight distribution occurred in the case of the comparison of the values of the ultrasonic sieve analysis with those of Diffraction Spectrometer 2. Here, only the 3C powder is out of place – a measuring error must be assumed. The correspondence of the other curves suggests that this diffraction spectrometer was calibrated after a sieve analysis.

The results of the microscopic investigations can be compared very poorly with the other results. In general, the passages are considerably lower here, irrespective of the particle form. Deviations from the other measuring processes were shown, particularly in the case of small particles. One cause is possibly the measurement of shadow images because, if the transmitted light is not parallel, the shadows may be larger than the particles casting them. However, it would also be conceivable that the microscopes do portray the real distribution. In fact, this should be assumed in the case of particles with a spherical form. However, this would mean that the sieve analyses and the processes which were calibrated according to these generally portray the particles as being too fine

## 7 Concluding remarks

The two processes whose diameter definition is not based on physical principles (microscopy and sieving) exhibit very distinct differences in the results. Even in the case of the measurement of spherical particles where microscopy should provide particularly reliable values, the proportion of extremely small particles deviated from the sieve analysis. In the microscope, the powders are portrayed as being larger than they actually are. Even with the aid of the two other measuring methods (diffraction spectroscopy and sedimentation), it cannot be proven with certainty that microscopy portrays the size distribution incorrectly since both methods determine the equivalent diameters by means of conversion and this procedure is influenced by their software. The important factors include according to what the devices were calibrated.

The results of the investigation show that no general conversion factor is possible between the individual measuring processes since even those devices operating according to the same physical principle may provide extremely divergent results. Therefore, it is not permissible to compare grain sizes or grain-size distributions which were determined using different methods.

However, the representation of the cumulative passage distribution permits a tendency-related statement to be made as to whether one method portrays a powder as being finer or coarser than another method. Therefore, the different processes only provide reference values for the characterisation of the powder quality. Although there are deviations in the results, all the methods are nevertheless of technical benefit. In this respect, the rapidity of determination is one point in favour of measurement using a diffraction spectrometer and the widespread application of the sieve analysis is one factor in its favour.

Fig. 1. Particle-size distribution determined with different methods of analyses; 1C, 1M, 1F (spherical) powders.

Durchgang	passage
Partikelgröße	particle size
Pulver	powder
Beugungsspektrometer	diffraction spectrometer
Sedigraph	sedigraph
Sieb / Ultraschallsieb	sieve / ultrasonic sieve
Mikroskop	microscope

Fig. 2. Particle-size distribution determined with different methods of analyses; 2C, 2M, 2F (spherical) powders.

Durchgang	passage
Partikelgröße	particle size
Pulver	powder
Beugungsspektrometer	diffraction spectrometer
Sedigraph	sedigraph
Sieb / Ultraschallsieb	sieve / ultrasonic sieve
Mikroskop	microscope

Fig. 3. Particle-size distribution determined with different methods of analyses; 3C, 3M, 3F (blocky/angular) powders.

Durchgang	passage
Partikelgröße	particle size
Pulver	powder
Beugungsspektrometer	diffraction spectrometer
Sedigraph	sedigraph
Sieb / Ultraschallsieb	sieve / ultrasonic sieve
Mikroskop	microscope

Fig. 4. Particle-size distribution determined with different methods of analysis; 4C, 4M, 4F (spattered) powders.

Durchgang	passage
Partikelgröße	particle size
Pulver	powder
Beugungsspektrometer	diffraction spectrometer
Sedigraph	sedigraph
Sieb / Ultraschallsieb	sieve / ultrasonic sieve
Mikroskop	microscope