The research methodology of powders with particle size of under 500 microns

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Abstract: *Presented results in this article are illustration of a methodology, which is the subject of this work. It contains a description of the main methods of investigations as microscopic observations, sieve analysis or examination of chemical composition by using EDX detector. The results present the determination of shape of particles, determination of size of particles and selections of fractions with the largest percentage and determination of chemical composition. The main goal of article is to present the order of studies of powders with a particle size of under 500 μm. Presented studies were carried out on iron powder with the particle size less than 250 μm. This powder is used for the production of metal cored electrodes for welding. Powder of iron is used as a filler in these electrodes.*

Keywords: *Methodology, tubular electrode, powders*

Metodologia badań proszków o wielkości cząstek poniżej 500 mikronów

Streszczenie: *Wyniki przedstawione w artykule są zobrazowaniem metodologii będącej tematem tej pracy. Zawiera ona opis głównych metod badań jak obserwacje mikroskopowe, analiza sitowa oraz określanie składu chemicznego z wykorzystaniem detektora EDX. Wyniki przedstawiają określenie kształtu cząstek, określenie wielkości cząstek wraz z wyróżnieniem frakcji o największym udziale procentowym oraz określenie składu chemicznego. Celem artykułu jest przedstawienie kolejności wykonywania badań dotyczących proszku żelaza o granulacji do 250 mikrometrów. Badania te można również z powodzeniem zastosować do określania wymienionych wielkości proszków o granulacji do 500 mikrometrów.*

Słowa kluczowe: *metodologia, druty proszkowe, proszki*

1. Introduction

Research methodology is necessary knowledge which helps researches to go from thought to action. It determinates work time, results and its quality. Effects of investigations are more clearly due to methodology.

The main goal is to present basic research of powders with particle size of under 500 μm. They are based on norms, literature and experience.

In the article, an expression of fraction is often appearing term. Referring to the content of tests, fraction should be understood as a part of a larger whole, which is obtained from the separation of the tested material. The possibility of obtaining the fractions is determined by various physical properties of these material. Referring to tests determining the particle size distribution, that feature is particle size [1].

Another slogan is the nominal size of the holes in sieves. This is a term that describes the size of mesh, which is part of the sieves used in the sieve analysis.

It should be also paid attention to equivalent terms which are appearing in the article. The percentage shares of each fraction (obtained by resolution of the tested material) is equivalent to the presentation of particle size distribution.

The article presents research methodology of powders with a particle size of under 500 microns. In this study, the methods which have enabled determination of particle shape, particle size distribution and chemical composition. Among possibilities to perform the analyzes a particle size sieve analysis was used.

2. Materials and methods

2.1. Description of investigation

The subject of research was iron powder with a grain size of less than 250 μm. It is used as one of the main components of the metal cored electrodes for welding. It serves as the filler of these electrodes. The powders which are used for composition of the core of metal cored electrodes meet a number of requirements including: the shape of the powder particles, the particle size, the percentage of the fractions, and an appropriate chemical composition. Their suitable parameters will provide proper fulfillment of the core of metal cored electrode and the properties of the welded joints.

To determine these properties, a well known methods of research, such as microscopic observation of the powder in a free-flowing state, the tests of chemical composition and particle size analysis are used.

2.2. Microscopic observations

The first step is to conduct microscopic examination of the powder in a flowing state. This study allows to specify particles of the smallest and largest sizes. These values are necessary for further choice corresponding to nominal holes size in sieves, and thus adequate quantities of sieves needed to perform the sieve analysis. In addition, microscopic observation of the powder in a flowing state help to determine the shape of the occurring particles according to PN-EN ISO 3252: 2002. Determination of particle size and particle shape was made by using a microscope Phenom G2 at a magnification of 420x.

2.3. Particle size analysis

The second step is to analyze the distribution of particle size. This can be done by using one of several available methods. Among them the most popular are: sieve analysis, analysis of laser and dynamic image analysis. Each of these methods has its own characteristics which determine its use. In each of the methods, the results may determine the percentages of the various fractions obtained during the tests. In addition, dynamic image analysis can be used in determining the particle size distribution, also allows for the assessment of their shape. During the choosing of the tests methods, it should be remember that each of the presented possibilities based on different phenomena. In view of the above, the received results from different methods should not to be compared directly [2]. However there is a possibility of attempting to confront results by using appropriate algorithms. The ranges of the size of particles in fraction in sieving analysis are dependent on the nominal mesh size in used sieves and their quantities. In turn, in the laser method the ranges are optimally matched to the geometry of the detector and the optical configuration [3]. One of the next differences between these methods is the required amount of material for the tests. In the sieve analysis it is necessary to have about 100 cm^3 [4] (100 cm^3 of iron powder is approximately 362 g) and the analysis with the laser needs just 1 g of powder. In addition, you can also select the fractions with the highest percentage occurring in the powder. The article presents the results of dry sieve analysis. Important parameters during the analysis are the right time and the amplitude of the shaking. These values are determined experimentally. On the basis of carried out analysis of the powders of metals and minerals, and mixtures thereof, was found that the powders which are dusty should be shaking at a lower amplitude. This can reduce the losses of material during the test. Sieve analysis was performed by using:

- vibratory shaker FRITCH model Analysette 3 PRO,
- set of sieves FRITCH of the nominal mesh size of: 20, 40, 50, 56, 63, 71, 80, 100, 125, 160, 180, 200 i 250 μm,
- laboratory weight RADWAG model: WPS 1200/C/2 with an accuracy of 0.01g,
- ultrasonic ULTRON U-24 cleaner; washing parameters: wash time was 10 minutes with vibration frequency of 21.5 kHz and wash temperature of 28°C.

2.4. Analysis of the chemical composition

The next step is to perform a study to determine the chemical composition. That analysis allows to identify the specific existing elements with their percentage and atomic and compounds that can combine with these elements. Before analysis, iron powder was inundated in resin, and its surface was sprayed with a layer of amorphous carbon. The tests were conducted by using a scanning electron microscope JEOL JSM 6610 with X-ray microanalyzer. Accelerating voltage of 20kV was used for analysis. A series of point studies were carried out to allow determination of the chemical composition of individual particles.

The results of microanalysis were recorded in the form of graphs of power spectrum of X-ray radiation which were analyzed quantitatively using ZAF method correction.

3. Results

3.1. Results of microscopic observations

Microscopic observation of the powder in a flowing state allowed to define the shape of the particles in accordance with PN-EN ISO 3252: 2002. Two visible characteristic shapes of particles, i.e. globular and granular were observed (fig. 1). Based on the analysis of particle size, it was

determined that the maximum size of particles was 300 µm. From this, 250 µm as a the maximum nominal mesh size of sieves was chosen. Therefore, 13 sieves in nominal mesh size from 20 µm to 250 µm were used in a sieve analysis. The quantity of used sieves depends on the accessible sieves. It also determinate the quantity of received compartments.

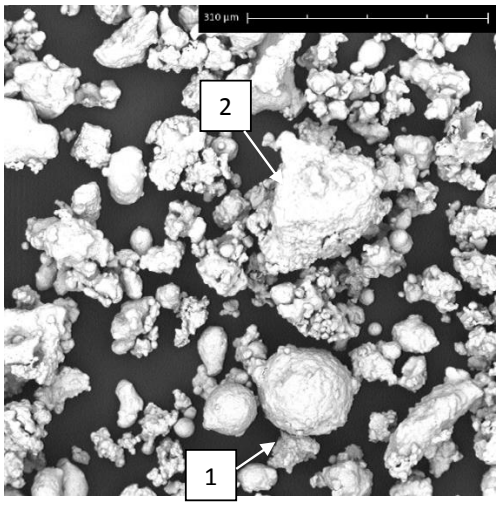

Fig. 1. SEM microstructure of iron powder with visible globular (1) and granular (2) particles.

3.2. Results of particle size analysis

Dry sieving analysis was performed with an amplitude of 1.5 mm for 5 minutes. The parameters values were selected basing on the experience. Recorded results of weighting the sieves before and after analysis were presented according to standard [4]. The table 1 shows the analysis parameters and the exact characteristics of the used equipment including the shape and size of sieves, the shape of the mesh or shaking method.

Based on these results, the dependence of the percentage sieve fraction as a function of nominal mesh size is determined (figure 2).

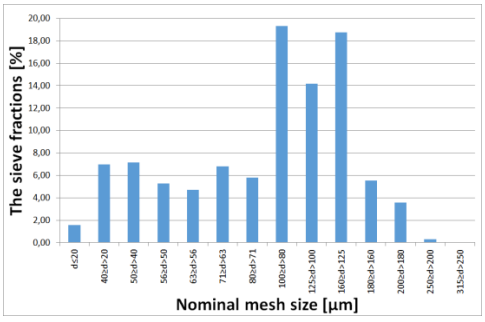

Fig. 2. Percentage participation of sieve fractions from iron powder

The results allowed to define the fractions, which participation in the powder was the greatest. It was found that the fraction with particle size from 80 to 160 µm constitute a majority, and their percentage participation is 52%.

Tab. 1. Results of sieving analysis of iron powder

Material:	Powder 1			
Method of sieving:	Dry / ~~Wet~~			
The size [mm] and shape o the sieve:	200			
	Round / ~~Squared~~			
Sieving element:	Woven wire / ~~Perforated sheet~~ / ~~Electrochemically perforated sheet~~			
Signage sieve:	~~Manually~~ / mechanically			
Type:	xyz			
The shape of the mesh:	~~Round~~ / squared			
Time of sieving [min]	5			
Amplitude [mm]	1.5			
1	**2**	**3**	**4**	**5**
Grain size	**The sieve fractions**		**Nominal mesh size**	**Collective grain**
µm	**g**	**%**	**µm**	**%**
315≥d>250	0.14	0.04	250	99.96
250≥d>200	1.13	0.31	200	99.65
200≥d>180	12.96	3.58	180	96.07
180≥d>160	20.1	5.56	160	90.51
160≥d>125	67.77	18.74	125	71.77
125≥d>100	51.22	14.16	100	57.60
100≥d>80	69.87	19.32	80	38.28
80≥d>71	20.92	5.78	71	32.50
71≥d>63	24.63	6.81	63	25.69
63≥d>56	17.02	4.71	56	20.98
56≥d>50	19.09	5.28	50	15.70
50≥d>40	25.89	7.16	40	8.54
40≥d>20	25.17	6.96	20	1.58
d≤20	5.72	1.58	The final collective grain	
Sum	**361.63**	**100**		
Output mass:	**361.91**			
Sum of the fractions mass:	**361.63**			
Losses:	**0.28 (0.08%)**			

3.3. Result of analysis of the chemical composition

Analysis of chemical composition confirmed that the particles compose of 100% iron. Places of analysis performance are shown in figure 3. They are consecutively designated by numbers 1, 2 and 3. Despite the diversity in the shape (globular and granular) of the occurring particles, it has no

influence on the chemical composition and they consisting in 100% iron (measured from 1 to 3). The results of the analysis of chemical composition are given in table 2. Energy spectrum of these measurements (figs. 4-6) is also included in the article.

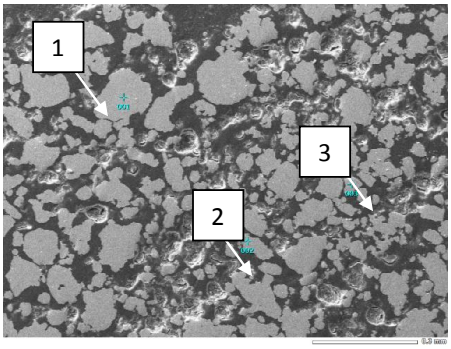

Fig. 3. SEM microstructure in cross-section of iron powder and marked the places of the analyzes of chemical composition.

Tab. 2. Results of analysis of chemical compositions

Number of measurement	Element	Mass participation [%]	Atomic participation [%]
1	Fe	100	100
2	Fe	100	100
3	Fe	100	100

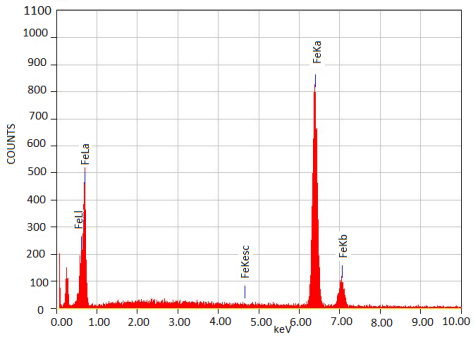

Fig. 4. X-ray energy spectrum obtained from a globular shaped particles (measurement 1)

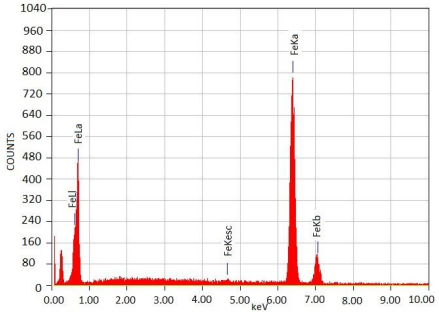

Fig. 5. X-ray energy spectrum obtained from a granular shaped particles (measurement 2)

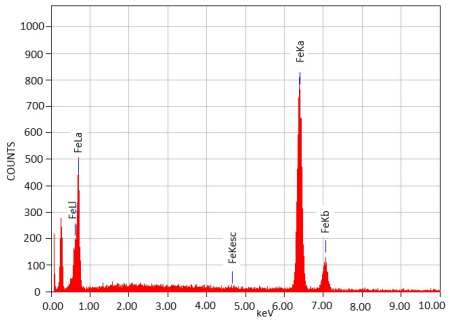

Fig. 6. X-ray energy spectrum obtained from a particle (measurement 3)

5. Conclusions

The presented methodology is a reflection of repeatedly performed tests powders of metals and minerals and their mixtures. It should contain microscopic observations using SEM, particle size analysis, where should be chosen the most appropriate method and analysis of the chemical composition using SEM with EDX detector. Presented course of action is a summary and attempt of methods for testing powders. Additionally, it should be received as a suggestion to the researches as well as the used methods.

The presented studies can be extended, for example by micro-hardness measurements or performance analysis of particle size distribution using all available methods and taking the trial of comparison each other.

References

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[3] Basic instructions Mastersizer "Introductory instructions - software 2.1 and later"
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