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Original scientific paper

The effect of the particle shape and structure on the flowability of electrolytic copper powder. II. The experimental verification of the model of the representative powder particle

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Abstract: An analysis of the effects of the shape, surface structure and size distribution of particles on the flowability of the copper powder was performed. It is shown that the most important property of the particles of a powder, regarding the flowability of the powder, is the surface structure of the particles.

Keywords: copper powder flowability, surface structure of particles of a flowing powder.

INTRODUCTION

It was pointed out in the previous paper¹ that the best flow of a copper powder can be expected if the particles of the powder are spherical and monosized, having a surface structure approaching that of a smooth metal relative to the jamming of the particles. The aim of this work was to show that the best flow is really reached if the above conditions are fulfilled and to determine which of the properties of powder particles is the most important in respect to the flowability of the powder.

EXPERIMENTAL

The experiments are performed as described in a previous study.² The apparent density and the flow rate (flowability) of a copper powder were determined as follows.

Determination of apparent density (Absolute Gravity) (ISO 3923/1-1979)

The method is intended for metallic powders that flow freely through a 2.5 mm diameter orifice. It may, however, be used for powders that flow with difficulty through a 2.5 mm diameter orifice but flow freely through a 5 mm diameter orifice. (If the powder does not flow, it is allowable to attempt to initiate flow by poking once with a 1 mm wire from the top of the funnel).

Measurements of the mass of a certain quantity of powder (the test sample shall be of at least 100 cm³

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volume to allow the determination to be carried out on three test portions), which in a loose condition exactly fills a cup of known volume (a capacity of $25 \pm 0.05 \text{ cm}^3$). The ratio between the mass and the volume represents the apparent density and is expressed in g/cm^3 .

Determination of flow rate (flowability) (ISO 4490-1978)

This standard is a method for the determination of the flow rate of metal powders and is suitable only for those which flow unaided through the specified apparatus. The flow rate is the speed at which a powder flows through orifices owing to gravity. To assess the speed, standardized funnels with varying calibrated openings are used (2.5 mm ϕ in the Hall flowmeter; 5.0 ϕ in the Carney flowmeter).

A certain amount of powder is poured in the funnel (50 ± 0.1) g and the flow time is recorded.

The test specimen should be kept at a temperature of 102 to 107 °C, for one hour in a drying oven and cooled to room temperature in a desiccator. Immediately prior to running the test, the specimen is removed from the desiccator and weighed. The discharge orifice, at the bottom of the funnel, is closed by placing a finger under it and the specimen transferred to the funnel, care being taken that the short stem of the funnel is filled. A stopwatch is started simultaneously with the removal of the finger from the discharge orifice and stopped at the instant the last of the powder leaves the funnel. The lapse of time in seconds is recorded.

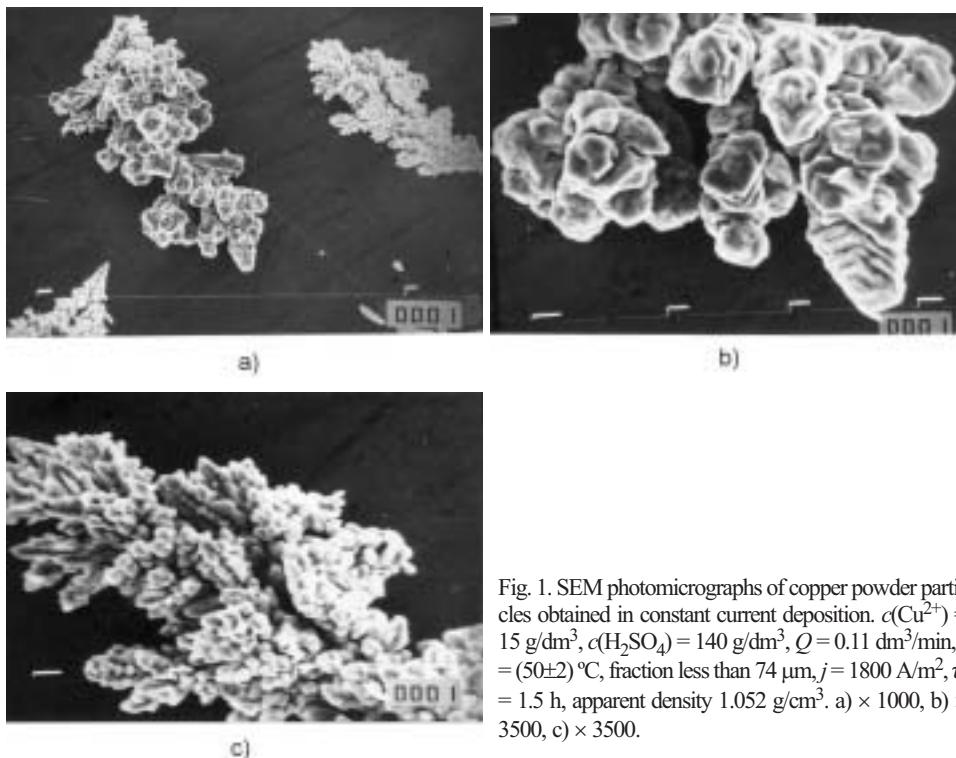


Fig. 1. SEM photomicrographs of copper powder particles obtained in constant current deposition. $c(\text{Cu}^{2+}) = 15 \text{ g/dm}^3$, $c(\text{H}_2\text{SO}_4) = 140 \text{ g/dm}^3$, $Q = 0.11 \text{ dm}^3/\text{min}$, $t = (50 \pm 2) \text{ }^\circ\text{C}$, fraction less than $74 \text{ }\mu\text{m}$, $j = 1800 \text{ A/m}^2$, $\tau_T = 1.5 \text{ h}$, apparent density 1.052 g/cm^3 . a) $\times 1000$, b) $\times 3500$, c) $\times 3500$.

RESULTS AND DISCUSSION

It is obvious that the shape and the structure of the powder particles are more important than the particle size distribution, when the flowability of a powder is considered. This is because there are non-sieved powders which can flow.

The structure of copper powder particles can be roughly divided into globular and dendritic. The first structure appears at lower, and second one at larger deposition overpotentials

or current densities.³ At the same time, the globular particles are more regular than the dendritic ones. The fraction $< 74 \mu\text{m}$ of copper powder, obtained at 1800 A/m^2 (powder A), flows and the typical particles of this fraction are shown in Fig. 1. Two different kinds of particles can be seen in Fig. 1, but in both cases the particles consist of globulae, positioned so

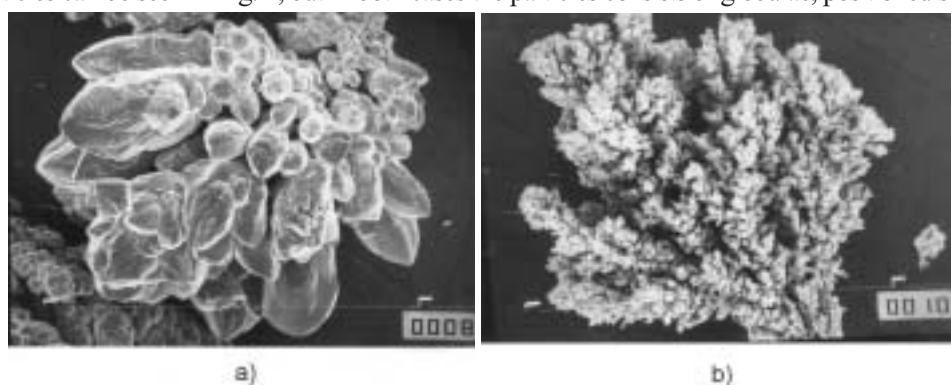
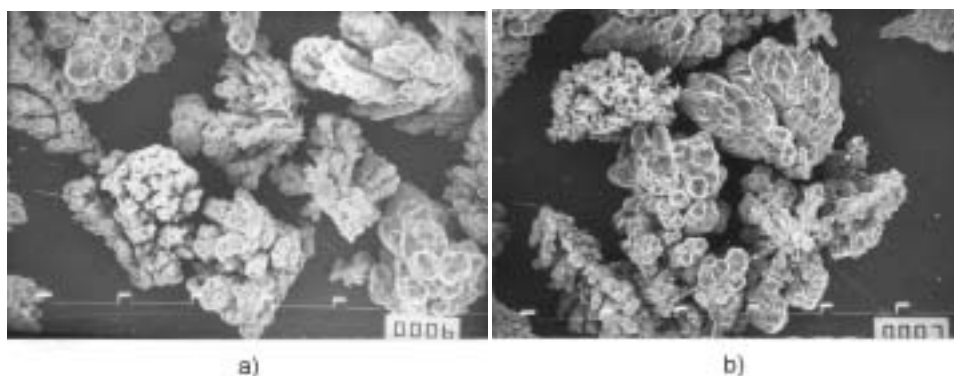


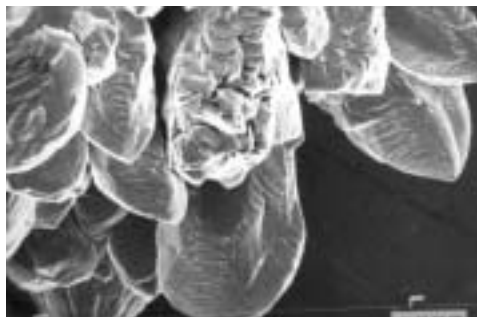
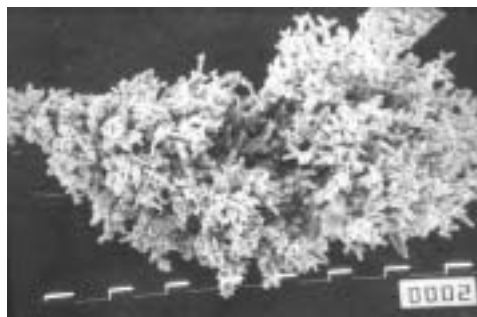
Fig. 2. SEM photomicrographs of copper powder particles obtained in constant current deposition. $c(\text{Cu}^{2+}) = 15 \text{ g/dm}^3$, $c(\text{H}_2\text{SO}_4) = 140 \text{ g/dm}^3$, $Q = 0.11 \text{ dm}^3/\text{min}$, $t = (50 \pm 2) \text{ }^\circ\text{C}$, fraction $(149-177) \mu\text{m}$, $\times 500$ (a) $j = 1800 \text{ A/m}^2$, $\tau_r = 1.5 \text{ h}$, apparent density 1.122 g/cm^3 , (b) $j = 3600 \text{ A/m}^2$, $\tau_r = 15 \text{ min}$, apparent density 0.524 g/cm^3 .



close to each other, that the interweaving of particles is not possible. It should be noted that the flow time in this case is about 40 s, which corresponds to the upper limit of the good flow of copper powder or the lower limit of the poor flow.² This is probably due to the irregular shape of the powder particles. This also means that the surface shape is less important for the flowability of powder than the surface structure of the particles.

This surface structure of the powder particles, which does allow jamming, is illustrated schematically by Figs. 2a, 2b and 3 from the previous paper.¹ This happens¹ when the surface parts of the particles corresponding to the metal segments are larger than, or equal to the pores between them.

Typical particles of the fraction $149-177 \mu\text{m}$ of powders A and B are shown in Fig. 2. (Powder B was obtained at 3600 A/m^2). The fraction of powder A exhibits excellent

Fig. 4. The same as in Fig. 2a, but $\times 1000$.Fig. 5. The same as in Fig. 2b, but $\times 1500$.

flowability, while the fraction of powder B does not flow. The shape of both particles can be taken as spherical, to the first approximation, but the surface structure is very different.

This is further proof that the surface structure of particles determines the flowability of a powder. It can be seen from Fig. 3 that the particles of powder A cannot interweave. Besides, the association of the particles from Fig. 3 is an excellent illustration of the model of a flowing powder as developed in the previous paper.¹ The particles are almost monosized and spherical, and their surface structure, regardless of being different from particle to particle, corresponds to the ones from Figs. 2a, 2b and 3 from the previous work.¹ The flow time of fraction 149–177 μm of powder A was about 20 s, which corresponds to excellent flowability.² Both fractions of powder A flow, but the non-sieved powder does not flow. This can be explained by Figs. 1a, 2a, 3 and 4. It is obvious that the particles from Fig. 1a can jam the particle from Figs. 2a and 4. In this way the particles from Fig. 3 can be abridged and the flow of the powder prevented. Hence, the surface structure of the particles of a non-sieved powder which can flow must be less rough than is the case of powder A. This probably characterizes powders with apparent density larger than 2.2 g/cm^3 .

On the other hand, the structure of the particle of fraction 149–177 μm of powder B from Fig. 2b is very porous and such particles can interweave as illustrated by Fig. 5. Obviously, such behavior, which leads to a non-flowing powder, is predicted by Fig. 2c of the previous paper.¹

Besides, the particles of fraction 149–177 μm of powder B can be structured as described earlier¹ by the slices model in Fig. 1 as can be seen in Fig. 6. This structure can also

Fig. 6. The same as in Fig. 2b, but $\times 500$.

lead to a non-flowing powder, but in this situation it is not of the practical significance, because all the surface of such a particle is suitable for jamming. Finally, it can be concluded, that the surface structure of powder particles is more important for the flowability of a powder than a regular particle shape, if poor flow is under consideration. If good flow of a powder is required, then a regular, compact, form of the particles is also required.

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ИЗВОД

УТИЦАЈ ОБЛИКА И СТРУКТУРЕ ЧЕСТИЦА НА ТЕЧЉИВОСТ
ЕЛЕКТРОЛИТИЧКОГ БАКАРНОГ ПРАХА. II. ЕКСПЕРИМЕНТАЛНА ПОТВРДА
МОДЕЛА РЕПРЕЗЕНТАТИВНЕ ЧЕСТИЦЕ ПРАХА

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Изведена је анализа утицаја облика, површинске структуре и расподеле величина честица на течљивост бакарног праха. Показано је да површинска структура представља најважније својство честице праха у односу на његову течљивост.

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