Processes of Multilayer Coatings Deposition Onto Powder Materials and Equipment for Such Processes

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ABSTRACT

The paper presents processes of multilayer deposition of refractory and fusible metals and alloys onto powder materials. The paper also presents evaluation of deposition conditions necessary to ensure high thickness uniformity of coating on a single powder particle as well as on entire powder mass. The paper presents experimental results of coating deposition, methods of removal of heat load, powder material transportation, deposition methods, and on-line control of coated material quality. The paper presents example of high-productive vacuum equipment for implementation of powder material coating technology.

INTRODUCTION

The problem of coating deposition in vacuum on disperses powder materials represents significant interest. There are known works describing metal coating deposition on disperse artificial diamond for maintenance of high adhesion of diamond grains with a bundle (sheaf) during manufacture of the cutting tool. In Sidrabe, Inc., vacuum machines for transportation and coating deposition on such type of materials have been developed and made [1].

Except for that on Sidrabe, Inc., vacuum machines for copper deposition on the graphite powder used for manufacture of electric brushes for high current transfer devices have been developed and made. Works on coating deposition on mica particles for creation of pigments are known [2]. High requirements to optical properties of the coated powders demand also high requirements to coating uniformity on each particle. The present work is devoted to possibility estimation to deposit homogeneous by thickness coatings on disperse powders and design of vacuum machines for realization of such process.

At coating deposition on powder materials there are a number of problems:

- aggregation of powders and impossibility of reception of a homogeneous thickness coating on each particle;
- maintenance of conditions for powder transportation for uniform coating of each particle on all surface.

In addition, we will consider a model of process of coating deposition on spherical particles (balls).

MODEL FOR CALCULATION

Balls are falling down from hopper and move downwards, filling itself in rectangular parallelepiped. The sizes of cross section or the parallelepiped basis can be treated as width *a* and thickness *b* for flow of falling balls in a zone of metallization. We admit that balls drop out of the hopper by layers with the same number of balls in any layer:

$$z * y = \left(b/d\right) * \left(a/d\right),$$

where *z* - number of balls in one row (determines or is determined by thickness of the flow b); *y* - number of rows in a layer of balls; *d* - diameter of a ball.

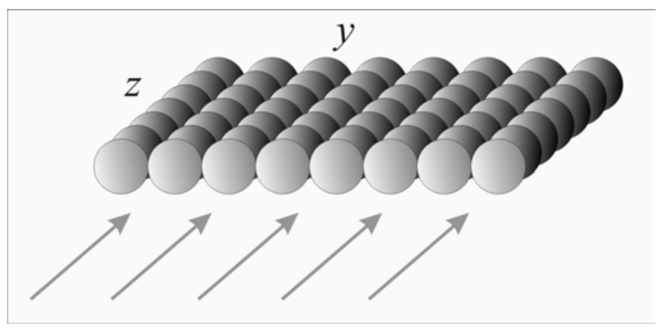

Figure 1. Configuration of ball layer.

Lets take one row of the balls and give a number to every ball: *i = 1, 2, 3 . . . z.*

Lets assume that going through the deposition zone, random arrangement of the balls in accented row (happened before entering deposition zone) is constant and metal is deposited on the balls that are closest to the evaporator, i.e. balls that are coated prevent deposition on other balls (*z* – 1). During the next run, the arrangement of the balls most likely will be different.

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Lets name standing of <u>*each*</u> ball in a most right position during traversal through deposition zone of *z* balls as <u>favorable event</u>. Probability of occurrence the favorable event during one run of accented row of z balls is:

$$p = 1 / z \, .$$

How we can treat the probability of the <u>favorable event</u> in other ways?

$p = 1 / z$ is a probability that 1/z part of all loaded balls gets metal during one run through the evaporation zone.

Lets assume that accented row of *z* balls goes through the evaporation zone *n* times, i.e. makes n runs. Lets find a probability $\mathbf{P}_{m,\,n}$ that after n runs the favorable event will take place exactly *m* times. Because standing of numbered balls during each run is random and independent event, we will use Poisson distribution in order to estimate probability *Pn(m)*, i.e. quantity of favorable events *m* during *n* runs of balls through the evaporation zone. Binomial distribution for discontinuous (discrete) eventual value at high *n* and low chance of favorable event *p* in each separate experiment (run in our case) is incorporated in Poisson distribution. This distribution can be described by formula:

$$P(m) = \frac{a^m}{m!} \cdot e^{-a}$$

where $a = p \bullet n$ - expectation (mathematical average).

MODELING RESULTS

By using Poisson formula, it is possible to make calculation *P(m)* and *R(m)* for four cases. Calculated values of $P_i(m)$ and $R_i(m)$ are shown in Table 1, Table 2 and in Figures 2 and 3.

Table 1. Conditions for calculations**.**

# of calc.	Number of runs	Amount of the balls in the row	Average of distribution *a*	Denotation of calculated values
1	2500	50	50	$P_1(m); R_1(m)$
2	2500	75	33	$P_2(m); R_2(m)$
3	2500	100	25	$P_3(m); R_3(m)$
4	2500	150	17	$P_4(m); R_4(m)$

$P_i(m)$ - probability value of that favorable event happens *m* times; $R_i(m)$ - probability value of that favorable event happens not less than *m* times.

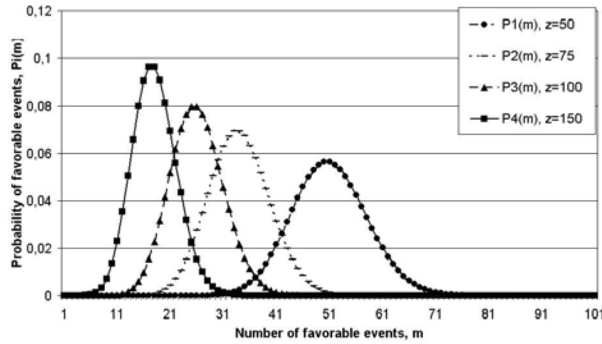

Figure 2. Series of favorable event probability distribution (Poisson formula) at different thickness of layer and same number of runs (n=2500).

Data of chart show that increase of layer thickness leads to decrease of the favorable events. By using Poisson formula, it is possible to make calculation *P(m)* and *R(m)* for four another cases:

Table 2. Condition for calculations.

# of calc.	Number of runs	Amount of the balls in the row	Average of distribution *a*	Denotation of calculated values
1	1000	75	13	$P_1(m); R_1(m)$
2	2500	75	33	$P_2(m); R_2(m)$
3	5000	75	66	$P_3(m); R_3(m)$
4	7500	75	100	$P_4(m); R_4(m)$

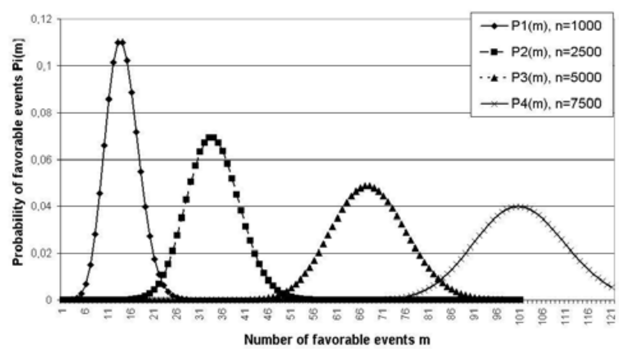

Figure 3. Series of favorable event probability distribution (Poisson formula) at different number of runs and same thickness of layer (z=75).

Data of Figure 3 show that growth of number of favorable events is proportional to increase of number of runs.

Let's consider a specific example of coating deposition on balls with diameter of 200 microns. We have flow of powder curtain from the hopper through a slot. Thickness of the slot can be adjusted. Width of the curtain is constant value -400 mm. At certain thickness of 15 mm, hopper's throat area will be 0,15 dm * 4 dm = 0,6 dm2. Initial speed of the balls at the moment of leaving the hopper is estimated as 0,5 m/sec. As a result, velocity is 0,6 dm2 * 5 dm/sec = 3 liters/sec. As per Bernoulli equation velocity should remain constant in the entire path of powder flow.

If it is loaded 120 liters of powder that is equivalent to 200 kg of powder and velocity of 3 l/sec, than complete turnover of the full mass of the powder is 40 seconds. Mass of the metals evaporated from the boats is about 10% of powder mass. Respectively, 20kg (10 kg from each boat) must be deposited onto the powder. Minimal evaporation rate is a limiting factor for deposition from boats. For the existing boats, this value is not less than 5 g/min. If vapor utilization ratio is 0,8 and evaporation rate 5 g/min, than deposition time for 10 kg is:

Considering that complete turnover of the full mass of the powder is 40 seconds, entire mass of the powder will make 3750 runs during evaporation time. Now we can review probability of favorable events at determined conditions.

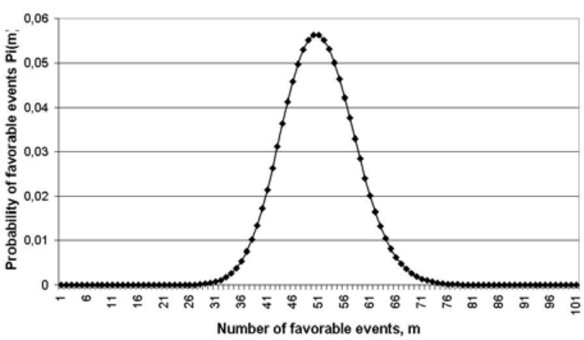

Figure 4. Series of favorable event probability distribution (Poisson formula) at curtain thickness 15 mm and 3750 runs of entire mass of the powder.

Analysis of these results shows that max of probability corresponds to 50 favorable events. Now, we will try to evaluate necessary number of runs in order to have uniformed coating on one ball. Area of ball surface zone with uniformity of ± 5% at one run is 1/20 of ball's surface. If we imagine ball as icosahedron, which equiprobable turns with new edge towards vapor source at each next run, it must mace at least 20 runs. Considering nonuniformity of thickness as ±10%, ratio of area of ball surface zone to ball's entire surface will be 10. In this case we have more favorable situation to get uniformed coating in 50 runs.

Now, please recall Figure 4. This calculation show that under conditions of 3750 runs, curtain thickness of 15 mm, probability that one ball gets into evaporation zone in edge position not less than 20 times is 0,9999988, i.e. has value close to 1 (Table 3).

Table 3

# of calc.	Number of runs	Amount of the balls in the row	Average of distribution *a*	$P_1(m)$	$R_1(m)$
1	3750	75	50	$P_1(20) = 7{,}57*10^{-7}$	$R_1(20) = 0{,}9999988$

Now, we will try to make calculation considering increase of effusion speed of flow out of the hopper. Lets assume that effusion speed is 1 m/sec. At the same geometrical parameters of the curtain, velocity of the balls will be 6 l/sec. If load is 120 liters that one run will take 20 seconds. This corresponds to number of runs equal to 7500. Now we can review probability of favorable events at determined conditions

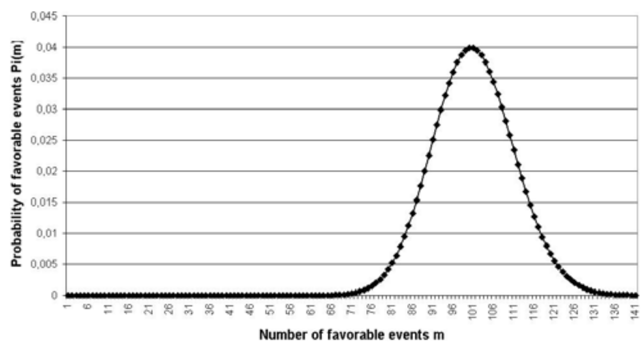

Figure 5. Series of favorable event probability distribution (Poisson formula) at curtain thickness 15 mm and 7500 runs of entire mass of the powder

In this case maximal probability corresponds to 101 favorable event. This calculation show that under conditions of 7500 runs, curtain thickness of 15 mm, probability that one ball gets into evaporation zone in edge position not less than 20 times is 1. (Table 4).

Table 4

# of calc.	Number of runs	Amount of the balls in the row	Average of distribution *a*	$P_1(m)$	$R_1(m)$
2	7500	75	100	$P_1(20) = 3{,}06*10^{-23}$	$R_1(20) = 1{,}0$

Finally, we would like to point out that accuracy of found results very much determined by correct choice of model and favorable event criteria. In this respect we tried to compare calculations with the data from our earlier conducted experiments.

EXPERIMENT

Some years ago we conducted experiments on powder coating by metal layer. Two batches of powder coated on our laboratory machine: #1 and #2 were statistically analyzed by the customer. We have those result and they, in our opinion, prove correctness of our approach. Tested batches of powder were exposed to metallic magnetron sputtering device in free-falling state. Metal was deposited from both sides of the curtain. Full load of powder in both cases was 50g or 188 cm^3. In both cases powder was in deposition zone about 300 times, i.e. had 300 runs. In both cases for "Chroma" measurements were taken random portions: #1 - 51 particle; #2 - 50 particles. Each particle was "Chroma" measured. The results of these measurements are stated below. "Chroma" is color characteristic, which is determined by thickness of coating from one side and uniformity of coating on particles from other side. In some ways this makes our cases closer! However we remember that "Chroma" growth stops after reaching of maximum and starts to decrease if deposition is continued. Lets hope that we will be on left side of curve *"Chroma"- time of metalizing*. For those two portions there was calculated such statistical parameters:

#1 Average value of "Chroma" - 47,22
Average standard deviation - 9,75
#2 Average value of "Chroma" - 43,37
Average standard deviation - 13,68

Moreover, average value of "Chroma" correlated with its integral value, calculated by us: #1 - 50,77; #2 - 47,87.

In Figure 6 you can see experimental and calculated functional relation of probability distribution of expected Chroma value (calculation was based on Poisson formula, with similar approximations as in previous calculations). By our mind, those findings prove correct choice of model for calculation of favorable event probability during deposition onto powders. On the basis of this modeling we designed industrial vacuum equipment for powder coating by one ore multilayer coating.

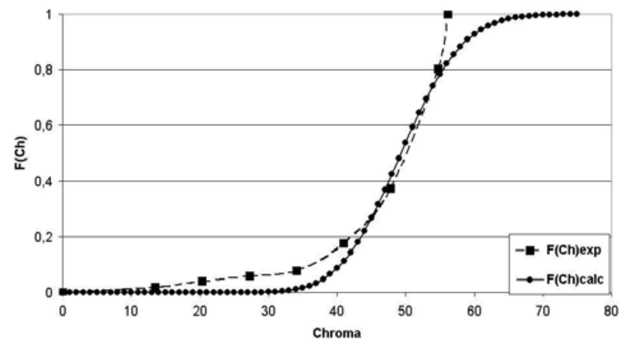

Figure 6. Functional relation of probability distribution (F(Ch)) for different value of Chroma.

CONCLUSIONS

The performed calculations proved correct choice of the model. On the basis of calculations there was proposed design of vacuum machine that can provide uniform coating of all powder mass with probability very close to 1.

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