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Design of experiments in thermal spraying: A review

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Abstract

The designs of experiments (DOE) methodology useful for thermal spraying and associated processes of post-spray treatment are thoroughly reviewed. The designs *Hadamard* (*Plackett–Burman*), two-level *full* and *fractional* factorial and also the response surfaces methodology are briefly described. The designs enable to obtain a polynomial regression equation which expresses the influence of process parameters on the response. The methods of determining of the significant coefficients of the regression equation (factors) are discussed. Examples of the application of different designs to determine the response equations with the responses related to microstructure, mechanical, electrical and other properties of coatings deposited using different thermal spray and post-spray processes are presented and discussed.

Keywords: Design of experiments; Parameters of thermal spraying processes

1. Introduction

Thermal spraying is the family of coating deposition processes in which molten, semi-molten or solid particles are deposited onto a substrate. The microstructure of the coatings results from their solidification and sintering [1]. The processes use hot gas, flame or plasma to accelerate the particles and to heat them up. Obtained coatings have a lamellar microstructure, which determines many of coatings properties. Since the optimization of coatings properties for a given specification needs a careful control of the operational spray parameters and the statistical methods can be useful in preparation of the experiments. The initial step in the design of experiments is a choice of variables being process parameters. In the simplest case, the parameters are fixed at low (−1) and high (+1) level. The experimental space is defined inside these parameters values. The choice of parameters needs some understanding of the process as there are as many as 50 process variables [2]. The first group of variables is related to the feedstock and concerns, supposing powder feedstock, mainly [3]:

- size distribution of powder;
- powder feed rate;
- powder morphology, such as e.g. shape, internal porosity, etc.

Typical variables for atmospheric plasma spraying are as follows:

- composition of working gas, such as e.g. vol.% of hydrogen in argon–hydrogen mixture;
- spray distance;
- powder feed rate;
- electric power input;
- carrier gas flow rate.

For high velocity oxy-fuel spraying, it is useful to add also another variable which is the ratio of fuel gas to oxygen [4] as well as other parameters for arc spraying, flame spraying or cold gas spraying. Another problem concerns pre-spray (sand blasting) and post-spray treatment (laser glazing, hot isostatic pressing, infiltration...), which also is frequently

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Table 1
Hadamard matrix for 7 variables

No. of experiment	X_1	X_2	X_3	X_4	X_5	X_6	X_7
1	−1	−1	−1	−1	−1	−1	−1
2	1	−1	−1	1	−1	−1	1
3	−1	1	−1	1	1	1	1
4	1	1	−1	−1	1	1	−1
5	−1	−1	1	−1	1	1	1
6	1	−1	1	1	1	1	−1
7	−1	1	1	1	−1	−1	−1
8	1	1	1	−1	−1	−1	1

Table 2
Another type of *Hadamard* matrix for 7 variables being also the *fractional* 2^{7-4} matrix for 7 variables

No. of experiment	X_1	X_2	X_3	$X_4=X_1X_2$	$X_5=X_1X_3$	$X_6=X_2X_3$	$X_7=X_1X_2X_3$
1	−1	−1	−1	1	1	1	−1
2	1	−1	−1	−1	−1	1	1
3	−1	1	−1	−1	1	−1	1
4	1	1	−1	1	−1	−1	−1
5	−1	−1	1	1	−1	−1	1
6	1	−1	1	−1	1	−1	−1
7	−1	1	1	−1	−1	1	−1
8	1	1	1	1	1	1	1

Three first columns express the *full* 2^3 matrix for 3 variables.

optimized. To take only one example, the typical variables for laser treatment include [5]:

- type of laser, i.e. emission wavelength;
- laser power density;
- scan velocity.

The properties of coatings sprayed in different experiments (responses) can be represented as a polynomial equation (regression equation) of following form:

$$Y = b_0 + \sum b_j X_j + \sum b_{ij} X_i X_j + \sum b_{ijk} X_i X_j X_k \quad (1)$$

Where i, j, k vary from 1 to the number of variables; coefficient b_0 is the mean of responses of all the experiment; b_i coefficient represents the effect of the variable X_i, and b_{ij}, b_{ijk} are the coefficients of regression which represent the effects of interactions of variables X_iX_j, $X_iX_jX_k$ respectively. The methods of the determination of the coefficients by using different designs of experiments are shortly outlined in the paper together with the methods of estimation of their signification. The experimental designs are described by taking the examples of thermal spray experiments realized and reported in the available literature.

2. Useful plans of experiments used in thermal spraying

Design of experiments has become a highly developed area with a number of textbooks which explain the backgrounds of the statistical methods [6–14]. A short compilation of most useful designs for the purpose of thermal spraying, based onto a literature review from this area will be discussed in this section. The papers were selected to find out the most typical ones and to cover a wide array the properties of thermally sprayed coatings. The compilation will be categorized into the following groups:

- *Hadamard* or *Plackett–Burman* [11] matrices;
- two-level *full* factorial designs (2^k);
- two-level *fractional* factorial designs (2^{k-m});
- Response of surface methodology (RSM) designs.

2.1 Hadamard (Plackett–Burman) matrix (screening matrix)

The *Hadamard* matrix is used to start the optimization by screening a great number of factors X_i, (i>4) that can potentially influence the response Y [10]. Each factor can take two levels (−1 or +1) in coded variables, corresponding to two levels of natural variables (say e.g. powder feed rate q_p=36 kg/h corresponds to X_i=−1 and powder feed rate of q_p=54.5 kg/h corresponds to X_i=+1). Tables 1 and 2 show the construction of two equivalent *Hadamard* matrices (called H_8) which make it possible to study of the effects of 7 factors (X_1 to X_7). The way of calculating the coefficient can be found in any

Table 3
Examples of *Hadamard*, two-level *fractional* and *Taguchi* design of experiments with the two-level factors used in thermal spraying

Number of experiments/type of plan	Spray technique	Spray material	Experimental factors	Tested responses	Reference
8/Hadamard	Air plasma spraying	Al_2O_3 and TiO_2	Arc current; primary gas flow; secondary gas flow; powder feed rate; spray distance; cooling	Porosity; hardness and microhardness; dielectric strength	[15]
8/Hadamard	High velocity combustion spraying	CrC–NiCr	Oxygen flow rate; propane flow rate; nitrogen flow rate; powder feed rate; gun barrel length; spray distance; substrate scan speed	Porosity, microhardness; tensile bond strength; pin-on-disk; abrasion wear	[16]
8/fractional 2^{6-3}	Water stabilized plasma	TiO_2	Scan speed; location; water flow; feed distance; powder feed rate; feed angle	Porosity; hardness and microhardness	[17]
16/Taguchi L_{16}	Air plasma spraying	$ZrO_2+Y_2O_3$	Cooling type; spray distance; spray angle; hydrogen flow rate; scan speed; current density; argon flow rate	Porosity, crack length; thermal conductivity	[18]