Materials and Design 55 (2014) 633-643

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Modeling the response of physical and mechanical properties of Cr–Mo prealloyed sintered steels to key manufacturing parameters



Materials & Design

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ARTICLE INFO

Article history: Received 5 January 2013 Accepted 12 October 2013 Available online 24 October 2013

Keywords: Powder metallurgy Cr–Mo prealloyed sintered steels Response surface methodology Physical and mechanical properties

ABSTRACT

In this study an experimental investigation using response surface methodology has been undertaken in order to model and evaluate the physical and mechanical properties of Cr–Mo prealloyed sintered steels with respect to the variation of powder metallurgy process parameters such as compacting pressure, sintering temperature and Cr content of the prealloyed steel powder. Mathematical models were developed at 95% confidence level to predict the physical properties such as sintered density and electrical resistivity and mechanical properties such as transverse rupture strength, apparent (=macro-)hardness, and impact energy. Analysis of variance was used to validate the adequacy of the developed models. The obtained mathematical models are useful not only for predicting the physical and mechanical properties with higher accuracy but also for selecting optimum manufacturing parameters to achieve the desired properties.

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1. Introduction

Powder metallurgy (PM) has proven to be an effective technique for manufacturing a variety of complex-shaped steel parts with accurate and reproducible dimensions, low cost, high performance and ability to be processed to net or at least near-net shape [1,2]. Higher relative density is one of the most important factors for producing high quality PM parts, since the density strongly influences the physical and mechanical properties [3–8].

To optimize a PM system, one of the important things to know is the relationship between the variables of interest, which are the optimization objects (e.g., the sintered density, impact energy and etc.) which were used (as typical) to evaluate some of the physical and mechanical properties, and the system factors, which are the key manufacturing parameters. The variables of the powder metallurgy process such as the technique and condition of compacting, the sintering sequences and also the type of powder, in particular the alloying technique [9] and alloy elements selected [10-12] strongly affect the physical and mechanical properties. There are several methods under development to increase the load bearing capacity of sintered steel parts. The major trends of these methods are the alloy development and/or modification of the manufacturing process. To keep the production cost low, single compaction as a relatively easy method compared e.g. to double or warm pressing is mostly employed for the preparation of the PM precision components. The desirable properties – within a given range – can then be achieved by controlling of the sintering procedure [8,13].

A large number of experimental investigations have been carried out relating manufacturing parameters of sintered steels to the properties, but only few of them substantiated their observations with a mathematical model. However, present day industrial application demands comprehensive theoretical simulation before actual design [13].

Response surface methodology (RSM) is a collection of mathematical and statistical techniques that is useful for the modeling, analyzing, and optimizing of objects in which a response of interest (typically a property) is influenced by several parameters [14–16]. RSM also quantifies the relationship between the controllable input parameters and the obtained responses (properties). The steps in this method involve: designing a series of experiments for adequate and reliable measurement of the response of interest, determining a mathematical model of the second-order response surface with the best fit, finding the optimal set of experimental parameters that produce a maximum or minimum value of response and representing the direct and interactive effects of process parameters through two and three dimensional plots [14,15].

In recent years, numerous researchers have used design of experiments (DOE) to analyze and model the key parameters for the PM technique [13,17–19]. Ji et al. [17] investigated the effect of sintering parameters such as sintering temperature, sintering time, heating rate and sintering atmosphere on the sintered density using the Taguchi method, based on orthogonal arrays (OA), which is widely used in research and industrial application.



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^{0261-3069/\$ -} see front matter @ 2013 Elsevier Ltd. All rights reserved. http://dx.doi.org/10.1016/j.matdes.2013.10.032

Table 1

Table 2

Symbols, levels and values of manufacturing parameters.

Symbol	Parameter	Unit	Level 1	Level 2	Level 3	Level 4	Level 5
А	Compacting pressure	(MPa)	250	400	500	600	700
В	Sintering temperature	(°C)	1120	1250	1300	-	-
С	Powder type	(wt.% Cr)	1.5	3	-	-	-

Tuble 2	
Design layout using the Design-Expert 8.0 software includi	ng experimental and predicted results for physical properties.

Standard order	Actual values of parameters			Sintered density (g cm ⁻³)		Electrical resistivity ($\mu \Omega cm$)	
	A	В	С	Actual values	Predicted values	Actual values	Predicted value
1	250	1120	1.5	6.11	6.12	27	27.71
2	400	1120	1.5	6.67	6.63	22	22.77
3	500	1120	1.5	6.85	6.87	21	20.73
4	600	1120	1.5	7.02	7.03	20	19.69
5	700	1120	1.5	7.12	7.11	20	19.65
6	250	1250	1.5	6.21	6.24	26	25.6
7	400	1250	1.5	6.74	6.73	22	21.29
8	500	1250	1.5	6.94	6.96	20	19.67
9	600	1250	1.5	7.11	7.11	20	19.05
10	700	1250	1.5	7.21	7.18	19	19.43
11	250	1300	1.5	6.35	6.34	23	23.49
12	400	1300	1.5	6.87	6.83	20	19.42
13	500	1300	1.5	7.04	7.05	18	17.96
14	600	1300	1.5	7.17	7.2	17	17.5
15	700	1300	1.5	7.26	7.26	17	18.04
16	250	1120	3	5.91	5.92	42	40.61
17	400	1120	3	6.5	6.47	33	33.85
18	500	1120	3	6.73	6.73	31	30.59
19	600	1120	3	6.9	6.92	28	28.33
20	700	1120	3	7.01	7.02	27	27.08
21	250	1250	3	6.01	6.04	38	38.1
22	400	1250	3	6.6	6.57	31	31.97
23	500	1250	3	6.82	6.83	29	29.13
24	600	1250	3	6.99	7	27	27.3
25	700	1250	3	7.12	7.09	26	26.46
26	250	1300	3	6.14	6.14	36	35.84
27	400	1300	3	6.68	6.67	29	29.95
28	500	1300	3	6.9	6.92	28	27.27
29	600	1300	3	7.07	7.09	26	25.6
30	700	1300	3	7.18	7.18	26	24.92

Bardhan et al. [13] established empirical relationships to predict the sintered density of ferrous powder compacts using second order response surface model based on central composite design (CCD). In another work, also Bardhan et al. [18] has used the CCD method for analyzing the surface roughness value of sintered iron PM components. The works of Davison and Selvakumar [19] focused on the two techniques, namely neural network (NN) and RSM for predicting the final density of sintered aluminum performs.

The objective of the present study is to use RSM in a full factorial design, to establish the functional relationships between three manufacturing key parameters of PM technique, namely alloying content of powder, compacting pressure and sintering temperature, with respect to sintered density, electrical resistivity, transverse rupture strength (TRS), apparent (=macro-)hardness and impact energy of Cr–Mo prealloyed sintered steels. These relationships can provide theoretical models based on empirical results that are used to analyze and predict the responses and to determine the optimal operating system parameters. The analysis of variance (ANOVA) shows that the experimental results fit well into the assumed RSM models.

2. Experimental procedures

Rectangular test specimens, $55 \times 10 \times 10 \text{ mm}^3$ according to DIN ISO 5754 [20] and $100 \times 12 \times 8 \text{ mm}^3$ in size were fabricated from prealloyed Astaloy CrL (Fe-1.5%Cr-0.2%Mo) and Astaloy

CrM (Fe-3%Cr-0.5%Mo) powders (supplied by Höganäs AB, Sweden), that were mixed with 0.6 and 0.5 wt.% C (natural graphite UF4), respectively, and microwax C as lubricant for a period of 60 min. Compaction was carried out uniaxially in pressing tools with floating die, and five compacting pressures were chosen (250, 400, 500, 600, 700 MPa) to obtain materials with different density levels. Then the green density was determined by measuring mass and dimensions. The green bodies were sintered at three different temperatures (1120 °C, 1250 °C, and 1300 °C). Sintering at 1120 °C was carried out in a SiC rod heated laboratory tube furnace (Type, "AHT") with gas-tight superalloy retort in flowing high purity nitrogen (5.0 grade = min. 99.999 purity, flow rate 2 l/min). To prevent of the sticking samples together, they were put in steel boxes filled with fused alumina granulate. Previously, delubing of these samples was accomplished in a small laboratory tube furnace (small AHT) at 600 °C for 30 min, and then the boat was pushed into the exit zone. After cooling, the boat was transported into the high temperature zone of the large AHT furnace and remained there for 60 min. For sintering at higher temperatures (1250 °C and 1300 °C), a pusher furnace with Mo heating coil on an Alumina muffle (Degussa type "baby" furnace) was used. To ensure reasonably clean atmosphere, the sintering was done in steel boxes filled with a mixture of 50 wt.% Al₂O₃ and 50 wt.% Fe-8%Al as getter. Delubing of these samples was done in the preheating zone of the furnace for 25 min, and then the boat was pushed into the high temperature (sintering) zone and remained there for 60 min. After

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