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Prediction of hardness for sintered HSS components using response surface method

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Abstract

Sintered high speed steel (HSS) components have been formed using powder metallurgy (PM) process. Water-atomized and vacuum—annealed powders of T-15 grade HSS along with other ingredients like Zn-stearate (2%) and alumina (Al_2O_3) were used to produce the components. The percentage of alumina, sintering temperature and sintering time were considered as the controllable process parameters while the hardness of the sintered components was considered as the response variable. A 2^3 full factorial design of experiments (DOE) was used to collect experimental data to statistically analyze the effect of process parameters on the hardness of sintered HSS components. It has been observed that the percentage of alumina, sintering temperature and also their interaction affects the hardness very significantly while duration of sintering temperature does not affects the hardness significantly. A second order response surface model (RSM) has been used to develop a predicting equation of hardness based on the data collected by a statistical design of experiments known as central composite design (CCD). The analysis of variance (ANOVA) shows that the observed data fits well into the assumed second order RSM model.

Keywords: Sintered components; Response surface; Central composite design; *t*-Test; *F*-Test

1. Introduction

Powder metallurgy (PM) HSS bar is well-established in Europe, USA and Japan as the main root of production of gear hubs, end mills, cold pressing dies and other high pressing tools. Titanium nitride and Titanium carbide coating processes have significantly increased tool life in many applications, while this has brought significant savings to the final customers. In the 1970s and early 1980s, massive rationalization took place world wide in the High Speed Steel Industries [1–8]. Tool users became more cost conscious and premature tool failures were no longer tolerated. It was realized several years before by the PM HSS components makers that with advances in conventional machining processes and the reduction in prices of conventional bar, component would only sell which had sufficient dimensional accuracy to avoid machining and which were totally repeatable in metallurgical quality. As a result the trend on PM HSS followed that of hard metals away from the large complex shaped components towards the index able inserts. It has been reported that 1–3% alumina addition during compaction enhances the tool life of the PM HSS cutting tool inserts.

In this study, an attempt has been made to develop a PM HSS cutting tool material with high hardness by improving the micro-structure as well as superior homogeneity with uniformly distributed carbides with uniform and finer grain size which results the PM HSS comparable with other hard metals like carbides in all respect [9–12]. Design of experiments (DOE) have been used to perform statistical analysis about the effect of various process parameters on the hardness of sintered HSS components and response surface method has been used to develop a predicting response surface equation for hardness of sintered HSS component.

2. Experimental procedures

The HSS powder of T-15 grade was supplied by M/S Hoganas Limited (Great Britain) and the chemical analysis was carried out by Powdrex in Great Britain. Leco Analyser and Hilger Polyvac were used for performing the analysis. The result of chemical analysis of T-15 grade HSS powder has been given in Table 1 (all data are in percentage of weight except where stated).

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Nomenclature

$\mathbf{B}_1$	$[\hat\beta_0 \quad \hat\beta_1 \quad \hat\beta_2 \quad \hat\beta_3 \quad \hat\beta_{12} \quad \hat\beta_{13} \quad \hat\beta_{23} \quad \hat\beta_{123}]^T$
$\mathbf{B}_2$	$[\hat\beta_0 \quad \hat\beta_1 \quad \hat\beta_2 \quad \hat\beta_3 \quad \hat\beta_{11} \quad \hat\beta_{22} \quad \hat\beta_{33} \quad \hat\beta_{12} \quad \hat\beta_{13} \quad \hat\beta_{23}]^T$
$E(x)$	mathematical expectation of the variable x
$F_{\text{estimated}}$	estimated value of Fisher's F-ratio
F_{α_s,ν_1,ν_2}	Fisher's F-ratio for ν_1 upper and ν_2 lower degrees of freedom for α_s level of significance
H	Hardness of sintered components
$\bar{H}$	Average value of hardness
$\bar{H}_i$	Average value of hardness for ith run number
$\bar{H}_{\text{oci}}$	Average value of hardness for central points
$\bar{\bar{H}}_{\text{c}}$	Average of averages of hardness values for central points
k	number of controllable process parameters
l	number of levels for each process parameter
m	number of coefficients in the regression equation
n_{a}	number of axial points = $2k$
n_{c}	number of central points
n_{f}	number of points used in factorial positions = 2^k
N	total number of design points = $n_{\text{f}} + n_{\text{a}} + n_{\text{c}}$
$t_{\text{estimated}}$	estimated t value
$t_{\alpha_s,\nu}$	value of Students t distribution for α_s level of significance and ν degrees of freedom
$\mathbf{X}$	a matrix formed by column vector $\mathbf{x}_0$, $\mathbf{x}_1$, $\mathbf{x}_2$, $\mathbf{x}_3$, ..., etc.
$\mathbf{X}^T$	transpose of the matrix $\mathbf{X}$
x_i	coded value of ith process parameter
$\mathbf{x}_0$	coloum vector of dummy variable i.e. column of 1's
$\mathbf{x}_i$	coloum vector of coded values for process parameter x_i
$\mathbf{x}_{ij}$	[scalar product of column vectors $\mathbf{x}_i$ and $\mathbf{x}_j$]
$\mathbf{x}_{ijk}$	[scalar product of column vectors $\mathbf{x}_i$, $\mathbf{x}_j$ and $\mathbf{x}_k$]
z_i	actual value of ith process parameter
$z_i^{\max}$	maximum actual value of the ith process parameter
$z_i^{\min}$	minimum actual value of the ith process parameter
z_i^0	centre point of the design or the basic level of the ith process parameter
Δz_i	unit or interval of variation on the z_i axis for the ith process parameter

Greek letters

α	distance from the centre point of the design to a star point (star arm)
β_0	free term of the regression equation
β_i	regression coefficient of ith process parameter (linear terms)
β_{ii}	regression coefficient of self interaction of ith process parameter (quadratic terms)
β_{ij}	regression coefficient of interaction between ith and jth process parameters (interaction terms)
β_{ijk}	regression coefficient of interaction among ith, jth and kth process parameters
$\hat\beta_0$	estimated value of β_0
$\hat\beta_i$	estimated value of β_i
$\hat\beta_{ij}$	estimated value of β_{ij}
$\hat\beta_{ii}$	estimated value of β_{ii}
$\hat\beta_{ijk}$	estimated value of β_{ijk}
ε	an error component
σ_e^2	estimate of error (replication variance)
σ_{res}^2	residual variance
σ_β^2	variance of regression coefficients

Table 1
Chemical Analysis of T-15 grade HSS powder

C	Co	Cr	V	W	Si	P	Mn	Mo	S	O (ppm)
1.605	5.03	3.92	4.82	12.02	0.36	0.01	0.23	0.8	0.018	733

Powder properties

Apparent density (gm/cm^3)	2.24
Flow (s/50 gm)	39.72
Compressibility (gm/cm^3)	5.96
Green strength (psi)	3059

Sieve distribution

Sieve number	Size (μm)	Cumulative (wt%)
+60#	>250	0.00
+85#	>180	0.00
+100#	>150	0.01
+150#	>106	9.29
+200#	>75	28.55
+350#	>45	62.15

The powder was compacted in a closed square die (as the shape of the square inserts) using 150 tonnes capacity hydraulic press. The die wall was lubricated with zinc stearate and the compacts were prepared according to a planned statistical design of experiments and the relative density of sintered performs were measured by hydrostatic process and the surface of the specimens was then polished with a fine emery paper. Hardness was determined by Rockwell Hardness tester using Scale B.

3. Effect of process parameters on hardness

In order to perform test of significance for individual process parameters as well as their interactions, an equation that can be considered is given by the following expression [14]:

$$\bar{H} = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{123} x_1 x_2 x_3 + \varepsilon, \quad (1)$$

and the corresponding fitted equation can be expressed as follows:

$$\begin{aligned}\hat{\bar{H}} &= E(\bar{H} - \varepsilon) \\ &= \hat\beta_0 + \hat\beta_1 x_1 + \hat\beta_2 x_2 + \hat\beta_3 x_3 + \hat\beta_{12} x_1 x_2 \\ &\quad + \hat\beta_{13} x_1 x_3 + \hat\beta_{23} x_2 x_3 + \hat\beta_{123} x_1 x_2 x_3.\end{aligned} \quad (2)$$

where $E(x)$ is the mathematical expectation of the variable x.

Table 2
Symbols, levels and values of process parameters

Process parameters (independent variables)	Symbols		Levels					
	Actual	Coded	Actual			Coded		
Binder (P + 2% ZS + alumina 2–4%)	z_1	x_1	2	3	4	−1	0	+1
Sintering temperature (°C)	z_2	x_2	1050	1150	1250	−1	0	+1
Sintering time (h)	z_3	x_3	1	1.5	2	−1	0	+1

Table 2 shows the parameter settings for performing statistical test on the degree of significance of process parameters and their interactions. For any factor z_i, the transformation from actual to coded values has been performed by considering Eqs. (3)–(5) given below [14]:

$$z_i^0 = \frac{z_i^{\max} + z_i^{\min}}{2}, \tag{3}$$

$$\Delta z_i = \frac{z_i^{\max} - z_i^{\min}}{2}, \tag{4}$$

$$x_i = \frac{z_i - z_i^0}{\Delta z_i}, \tag{5}$$

A full factorial experimental design (l^k) with six additional central points (n_c) has been considered for performing the statistical analysis. The six additional central points give an estimate of experimental error. Table 3 gives the observed data for different settings of process parameters. The data have been collected by conducting the experiments in a random order of run numbers and Eq. (1) has been fitted to the observed data by using MATLAB software (version 6.12). The coefficients of the fitted equations can be obtained from Eq. (6) given below [13].

$$\mathbf{B}_1 = (\mathbf{X}^{\mathrm{T}}\mathbf{X})^{-1}\mathbf{X}^{\mathrm{T}}\mathbf{H}_1, \tag{6}$$

where

$$\mathbf{B}_1 = [\hat{\beta}_0 \quad \hat{\beta}_1 \quad \hat{\beta}_2 \quad \hat{\beta}_3 \quad \hat{\beta}_{12} \quad \hat{\beta}_{13} \quad \hat{\beta}_{23} \quad \hat{\beta}_{123}]^{\mathrm{T}}$$
$$\mathbf{X} = [\mathbf{x}_0 \quad \mathbf{x}_1 \quad \mathbf{x}_2 \quad \mathbf{x}_3 \quad \mathbf{x}_{12} \quad \mathbf{x}_{13} \quad \mathbf{x}_{23} \quad \mathbf{x}_{123}],$$
$$\mathbf{x}_0 = [1 \quad 1 \quad 1 \quad 1 \quad 1 \quad 1 \quad 1 \quad 1]^{\mathrm{T}},$$
$$\mathbf{x}_1 = [-1 \quad 1 \quad -1 \quad 1 \quad -1 \quad 1 \quad -1 \quad 1]^{\mathrm{T}},$$
$$\mathbf{x}_2 = [-1 \quad -1 \quad 1 \quad 1 \quad -1 \quad -1 \quad 1 \quad 1]^{\mathrm{T}},$$
$$\mathbf{x}_3 = [-1 \quad -1 \quad -1 \quad -1 \quad 1 \quad 1 \quad 1 \quad 1]^{\mathrm{T}}$$
$$\mathbf{x}_{12} = [1 \quad -1 \quad -1 \quad 1 \quad 1 \quad -1 \quad -1 \quad 1]^{\mathrm{T}},$$
$$\mathbf{x}_{13} = [1 \quad -1 \quad 1 \quad -1 \quad -1 \quad 1 \quad -1 \quad 1]^{\mathrm{T}},$$
$$\mathbf{x}_{23} = [1 \quad 1 \quad -1 \quad -1 \quad -1 \quad -1 \quad 1 \quad 1]^{\mathrm{T}},$$
$$\mathbf{x}_{123} = [-1 \quad 1 \quad 1 \quad -1 \quad 1 \quad -1 \quad -1 \quad 1]^{\mathrm{T}},$$
$$\mathbf{H}_1 = [77.83 \quad 63.67 \quad 85.17 \quad 86.83 \quad 79.25 \quad 68.00 \quad 82.33 \quad 85.50]^{\mathrm{T}},$$

Hence, the fitted equations for H can be written as follows:

$$\hat{H} = 78.5725 - 2.5725x_1 + 6.385x_2 + 0.1975x_3 + 3.78x_1x_2 + 0.5525x_1x_3 - 1.24x_2x_3 - 0.175x_1x_2x_3 \tag{7}$$

Since the variance (covariance) matrix $(\mathbf{X}^{\mathrm{T}}\mathbf{X})^{-1}$ for a 2^3 full factorial experimental design is a diagonal matrix with each diagonal element $1/n_{\mathrm{f}}$, the coefficient of the regression equation are uncorrelated and hence all coefficients of Eq. (2) can be estimated with the same accuracy (σ_β). The Student's t-test

Table 3
Observed hardness-values for different settings of process parameters under 2^3 full factorial design

Run no.	Actual values of parameters			Coded values of parameters			Values of response variables (hardness) (Rockwell hardness, Scale B)						
	z_1	z_2	z_3	x_1	x_2	x_3	H_1	H_2	H_3	H_4	H_5	H_6	$\bar{H}$
1	2	1050	1	−1	−1	−1	77.5	75	76	83	77	78.5	77.83
2	4	1050	1	+1	−1	−1	65	66	61	60	66	64	63.67
3	2	1250	1	−1	+1	−1	78	85	88	83	88	89	85.17
4	4	1250	1	+1	+1	−1	84	86	89	83	88	91	86.83
5	2	1050	2	−1	−1	+1	78.5	81	82	73	81	80	79.25
6	4	1050	2	+1	−1	+1	70	70	68	65	67	68	68.00
7	2	1250	2	−1	+1	+1	80	85	80	80	85	84	82.33
8	4	1250	2	+1	+1	+1	82	87	88	83	96	87	85.50
9	3	1150	1.5	0	0	0	85	86	88	88	91	88	87.67
10	3	1150	1.5	0	0	0	86	79	91	85	91	90	87.00
11	3	1150	1.5	0	0	0	82	86	88	83	87	90	86.00
12	3	1150	1.5	0	0	0	84	92	91	88	88	87	88.33
13	3	1150	1.5	0	0	0	85	88	90	85.5	91	92	88.58
14	3	1150	1.5	0	0	0	80	85	87	82	84	83	83.50

can be used to perform statistical test of significance for the main effects as well as interactions of process parameters. The t-values for a particular process parameter or interaction can be obtained using the following equations [14]:

$$t_{\text{estimated}} = \frac{|\hat{\beta}_0 \text{ or } \hat{\beta}_i \text{ or } \hat{\beta}_{ij} \text{ or } \hat{\beta}_{ijk}|}{\sigma_\beta}, \tag{8}$$

$$\sigma_\beta^2 = \frac{\text{estimate of error}}{n_\text{f}} = \frac{\sigma_\text{e}^2}{n_\text{f}}, \tag{9}$$

where

$$\text{Estimate of error} = \sigma_\text{e}^2 = \sum_{i=1}^{n_\text{c}} \frac{(\bar{H}_{\text{oc}i} - \bar{\bar{H}}_\text{c})^2}{n_\text{c} - 1}, \tag{10}$$

For example, the estimated value of the coefficient of x_2 is 6.385. The observed values of $\bar{H}$ for six central design points (design points 9 to 14 of Table 3) can be used to estimate the values of σ_e^2, σ_β^2 and $t_{\text{estimated}}$ as follows:

$$\sigma_\text{e}^2 = \sum_{i=1}^{n_\text{c}} \frac{(\bar{H}_{\text{oc}i} - \bar{\bar{H}}_\text{c})^2}{(n_\text{c} - 1)} = 3.56463$$

$$\sigma_\beta^2 = \frac{3.56463}{8} = 0.445579$$

$$t_2 = \frac{(|\hat{\beta}_2|)}{\sigma_\beta} = 9.5652$$

Similarly, the t-values for other regression coefficients can be estimated as follows:

$$t_0 = 117.71, \quad t_1 = 3.8538, \quad t_3 = 0.2959, \quad t_{12} = 5.6627,$$

$$t_{13} = 0.8277, \quad t_{23} = 1.8576, \quad t_{123} = 0.2622$$

It can be seen from statistical table of Students t distribution that the standard t-values for 1 and 5% level of significance and 5 degrees of freedom ($\nu = n_\text{c} - 1 = 5$) are $t_{0.01;\,5} = 3.365$ and $t_{0.05;\,5} = 2.015$, respectively. From the estimated t-values of different coefficients it can be found that the binder (x_1), sintering temperature (x_2) and also their interaction ($x_1 \times x_2$) highly influence (significant at 1% level of significance) the hardness of sintered HSS components. Sintering time (x_3) and other interactions ($x_1 \times x_3$), ($x_2 \times x_3$), ($x_1 \times x_2 \times x_3$) do not affect the hardness significantly.

4. Modelling of hardness by response surface method

4.1. Response surface method

Response surface method (RSM) adopts both mathematical and statistical techniques which are useful for the modelling and analysis of problems in which a response of interest is influenced by several variables. RSM attempts to analyze the influence of the independent variables on a specific dependent variable (response). The independent variables denoted by $x_1, x_2, \ldots x_k$ are presumed to be continuous and can be controlled with negligible error. The response $\bar{H}$ is postulated to be a random variable. For two independent variables x_1 and x_2, the response $\bar{H}$ can be represented as a function of x_1 and x_2 as follows [13]:

$$\bar{H} = f(x_1, x_2) + \varepsilon, \tag{11}$$

'ε' represents an error component.

If the expected response is denoted by $E(\bar{H} - \varepsilon) = \hat{\bar{H}}$, then the surface represented by $\hat{\bar{H}} = f(x_1, x_2)$ is termed as the response surface. A second or higher order response surface model is necessary to approximate the surface around a curvature. In most cases, a second order response surface model is adequate which can be represented by the following equation [13]:

$$\bar{H} = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i}^{k} \sum_{j}^{k} \beta_{ij(i<j)} x_i x_j + \varepsilon, \tag{12}$$

and the fitted equation can be written as follows:

$$\begin{aligned} \hat{\bar{H}} &= E(\bar{H} - \varepsilon) \\ &= \hat{\beta}_0 + \sum_{i=1}^{k} \hat{\beta}_i x_i + \sum_{i=1}^{k} \hat{\beta}_{ii} x_i^2 + \sum_{i}^{k} \sum_{j}^{k} \hat{\beta}_{ij(i<j)} x_i x_j. \end{aligned} \tag{13}$$

4.2. Design of experiment and data collection

A second order response surface can adequately be represented by Eq. (13). In order to fit a second order model the experimental design must have at least three levels of each factor. An experimental design which is rotatable should be selected. A rotatable experimental design means that the variance of the predicted response $\hat{\bar{H}}$ at some point $\mathbf{x}$ (a vector of independent variables $x_1, x_2, \ldots, x_k$) is a function only of the distance of the point from the design centre and is not a function of direction [13]. An experimental design with this property will leave the variance of $\hat{\bar{H}}$ unchanged when the design is rotated about the design centre (0, 0, ..., 0). Any first order orthogonal design is rotatable. A 3^k experimental design and their fractions are not good choices for second order response surface model because the information surface and contours show that these experimental designs are not rotatable [15]. The central composite design (CCD) is the most widely used experimental design for modelling a second order response surface. A CCD consists of l^k factorial or fractional factorial points (usually coded ± 1 notation), augmented by $2k$ axial points $\{(\pm\alpha, 0, 0, \ldots, 0), (0, \pm\alpha, 0, \ldots, 0), (0, 0, \pm\alpha, \ldots, 0), (0, 0, \ldots, \pm\alpha)\}$ and n_c centre points (0, 0, 0, ..., 0). A CCD can be made rotatable by selecting the appropriate value of α and for a rotatable CCD, $\alpha = (n_\text{f})^{1/4}$. With proper choice of n_c, the CCD can be made orthogonal or it can be made uniform precision design [16]. The uniform precision design means that the variance of $\hat{\bar{H}}$ at origin is equal to the variance of $\hat{\bar{H}}$ at a unit distance from the origin. A uniform precision design ensures more protection against bias in the coefficients than an orthogonal design. Hence, a CCD with uniform precision has been selected for this study. The values of various parameters for our CCD with uniform precision are as follows [16]:

$$k = 3;\quad n_f = 8;\quad n_c = 6;\quad n_a = 6;\quad N = 20;\quad \alpha = 1.682;$$

Table 4 gives the parameter settings for fitting a second order response surface equation based on central composite design (CCD), while Table 5 gives the observed hardness values (responses) for different settings of process parameters. The data have been collected by conducting the experiments in a random order of run numbers and six replications for each design point have been taken.

5. Results and discussions

5.1. Response surfaces and interpretations

The second order response surface can be written as Eq. (13). The second order response surface equations have been fitted using MATLAB software (version 6.12). The coefficients of the fitted equations can be obtained from Eq. (14) given below:

$$\mathbf{B}_2 = (\mathbf{X}^{\mathrm{T}}\mathbf{X})^{-1}\mathbf{X}^{\mathrm{T}}\mathbf{H}_2, \tag{14}$$

where

$$\mathbf{B}_2 = [\,\hat{\beta}_0 \quad \hat{\beta}_1 \quad \hat{\beta}_2 \quad \hat{\beta}_3 \quad \hat{\beta}_{11} \quad \hat{\beta}_{22} \quad \hat{\beta}_{33} \quad \hat{\beta}_{12} \quad \hat{\beta}_{13} \quad \hat{\beta}_{23}\,]^{\mathrm{T}}$$

$$\mathbf{X} = [\,x_o \quad \mathbf{x}_1 \quad \mathbf{x}_2 \quad \mathbf{x}_3 \quad \mathbf{x}_{11} \quad \mathbf{x}_{22} \quad \mathbf{x}_{33} \quad \mathbf{x}_{12} \quad \mathbf{x}_{13} \quad \mathbf{x}_{23}\,]$$

$$\mathbf{x}_o = [\,1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1 \;\; 1\,]^{\mathrm{T}}$$

$$\mathbf{x}_2 = [\,-1 \;\; -1 \;\; 1 \;\; 1 \;\; -1 \;\; -1 \;\; 1 \;\; 1 \;\; 0 \;\; 0 \;\; -1.682 \;\; 1.682 \;\; 0 \;\; 0 \;\; 0 \;\; 0 \;\; 0 \;\; 0 \;\; 0 \;\; 0\,]^{\mathrm{T}},$$

$$\mathbf{x}_3 = [\,-1 \;\; 1 \;\; -1 \;\; 1 \;\; -1 \;\; 1 \;\; -1 \;\; 1 \;\; 0 \;\; 0 \;\; 0 \;\; 0 \;\; -1.862 \;\; 1.682 \;\; 0 \;\; 0 \;\; 0 \;\; 0 \;\; 0 \;\; 0\,]^{\mathrm{T}},$$

$$\mathbf{x}_{ij} = [\text{Scalar product of column vectors of } \mathbf{x}_i \text{ and } \mathbf{x}_j],$$

$$\mathbf{H}_2 = [\,77.83 \;\; 63.67 \;\; 85.17 \;\; 86.83 \;\; 79.25 \;\; 68.00 \;\; 82.33 \;\; 85.50 \;\; 81.50 \;\; 93.92 \;\; 94.08 \;\; 59.50 \;\; 91.17 \;\; 84.50 \;\; 87.67 \;\; 87.00 \;\; 86.00 \;\; 88.33 \;\; 88.58 \;\; 83.50\,]^{\mathrm{T}},$$

The experimental results given in Table 5 have been used to establish the second order response surface equation using MATLAB. The second order response surface model for hardness of sintered HSS is given by the following equation:

$$\hat{\hat{H}} = 88.6829 - 3.0363x_1 + 7.9984x_2 + 0.9371x_3 - 1.3963x_1^2 - 5.2562x_2^2 - 1.3521x_3^2 + 3.78x_1x_2 + 0.5525x_1x_3 - 1.24x_2x_3, \tag{15}$$

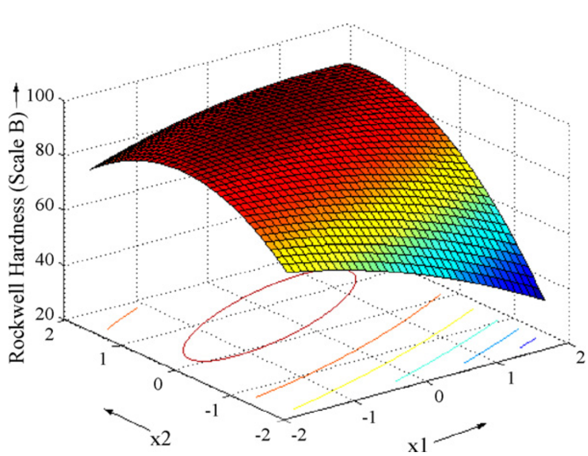

Fig. 1. Surface and contour plot for $\hat{\hat{H}} = f(x_1, x_2)$.

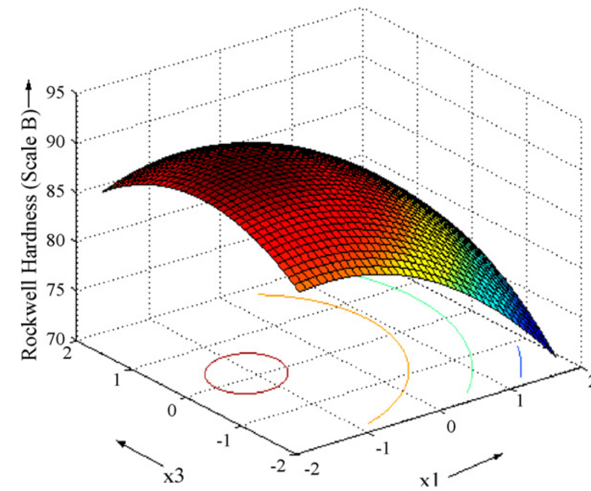

Fig. 2. Surface and contour plot for $\hat{\hat{H}} = f(x_1, x_3)$.

Figs. 1–3 gives the surface as well as the contour plots that show the dependence of hardness on the process parameters. The plots indicate the existence optimum values of process parameters for maximum hardness of sintered HSS components. The plots also show how hardness varies with the variation of values for process parameters. For example, Fig. 3 shows that hardness reduces rapidly if the sintering temperature is reduced below 1200 °C while only small variation in hardness is observed with the variation in sintering time.

5.2. Test of reliability for predicting response surface equation

Test of reliability for the predicting response surface equations has been carried out using both Fisher's variance ratio test and analysis of variance (ANOVA) as given below [14]:

Fisher's F-ratio test:

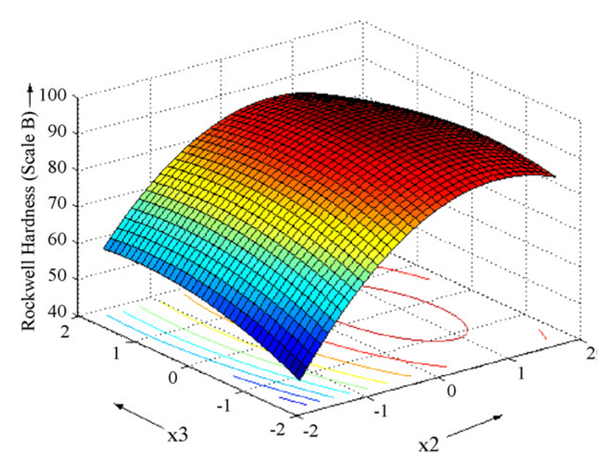

Fig. 3. Surface and contour plot for $\hat{\hat{H}} = f(x_2, x_3)$.

Table 4
Symbols, levels and values of process parameters

Process parameters (Independent variables)	Symbols		Levels									
	Actual	Coded	Actual					Coded				
Binder (%) (P + 2% ZS + alumina 2–4%)	z_1	x_1	1.381	2	3	4	4.682	−1.682	−1	0	+1	+1.682
Sintering temperature (°C)	z_2	x_2	881.8	1050	1150	1250	1418.2	−1.682	−1	0	+1	+1.682
Sintering time (h)	z_3	x_3	0.659	1	1.5	2	2.341	−1.682	−1	0	+1	+1.682

Table 5
Observed hardness data according to central composite design

Run no.	Actual values of parameters			Coded values of parameters			Values of response variables (hardness) (Rockwell hardness, Scale B)						
	z_1	z_2	z_3	x_1	x_2	x_3	H_1	H_2	H_3	H_4	H_5	H_6	$\bar{H}$
1	2	1050	1	−1	−1	−1	77.5	75	76	83	77	78.5	77.83
2	4	1050	1	+1	−1	−1	65	66	61	60	66	64	63.67
3	2	1250	1	−1	+1	−1	78	85	88	83	88	89	85.17
4	4	1250	1	+1	+1	−1	84	86	89	83	88	91	86.83
5	2	1050	2	−1	−1	+1	78.5	81	82	73	81	80	79.25
6	4	1050	2	+1	−1	+1	70	70	68	65	67	68	68.00
7	2	1250	2	−1	+1	+1	80	85	80	80	85	84	82.33
8	4	1250	2	+1	+1	+1	82	87	88	83	96	87	85.50
9	4.682	1150	1.5	1.682	0	0	83	82	84	78	79	83	81.50
10	1.318	1150	1.5	−1.682	0	0	90	96	93	92.5	95	97	93.92
11	3	1418	1.5	0	1.682	0	94	96	93	93	96.5	92	94.08
12	3	882	1.5	0	−1.682	0	64	65	69	51	53	55	59.50
13	3	1150	2.341	0	0	1.682	87	93	93	90	91	93	91.17
14	3	1150	0.659	0	0	−1.682	81	85	87	80	87	87	84.50
15	3	1150	1.5	0	0	0	85	86	88	88	91	88	87.67
16	3	1150	1.5	0	0	0	86	79	91	85	91	90	87.00
17	3	1150	1.5	0	0	0	82	86	88	83	87	90	86.00
18	3	1150	1.5	0	0	0	84	92	91	88	88	87	88.33
19	3	1150	1.5	0	0	0	85	88	90	85.5	91	92	88.58
20	3	1150	1.5	0	0	0	80	85	87	82	84	83	83.50

The Fisher's F-ratio is given by the following equation:

$$F_{\text{estimated}} = \frac{\sigma_{\text{res}}^2}{\sigma_{\text{e}}^2}, \tag{16}$$

where

$$\sigma_{\text{res}}^2 = \sum_{i=1}^{N} \frac{(\bar{H}_i - \hat{\bar{H}})^2}{(N-m)}, \tag{17}$$

and

$$\sigma_{\text{e}}^2 = \sum_{i=1}^{n_{\text{c}}} \frac{(\bar{H}_{\text{oci}} - \bar{\bar{H}}_{\text{c}})^2}{(n_{\text{c}} - 1)}. \tag{18}$$

If $F_{\text{estimated}} < F_{\alpha_{\text{s}};\nu_1,\nu_2}$ then the corresponding estimated regression equation fits the observed data adequately.

In our case,

$$\sigma_{\text{res}}^2 = 19.19461, \quad \sigma_{\text{e}}^2 = 3.56463 \quad \text{and} \quad F_{\text{estimated}} = 5.3847$$

Table 6
Analysis of variance for second order response surface equation

Source	DF	SS	MSS	$F_{\text{estimated}}$	$F_{\text{tabulated}}$	
					5% level	1% level
Regression	9	1460.71	162.30	9.26**	3.02	4.94
First order terms	3	1011.68	337.23	94.73**	5.41	12.06
Second order terms	6	449.03	74.84	21.02**	4.95	10.67
Residual Error	10	175.31	17.53			
Lack of fit	5	157.49	31.50	8.85*	5.05	10.97
Experimental error	5	17.82	3.56			
Total	19	1636.03				

$R^2 = 89.3\%$.
** Significant at both 1 and 5% level.
* Significant at 5% level.

The upper degrees of freedom ($\nu_1 = N - m$) and lower degrees of freedom ($\nu_2 = n_c - 1$) are 10 and 5, respectively. The F-value for 1% level of significance (for $\nu_1 = 10$, $\nu_2 = 5$) is $F_{0.01;\,10,\,5} = 10.05$. The estimated F-value for the predicting equation is much less than 10.05. Hence, it can be concluded that the established predicting equations gives an excellent fitting to the observed data.

5.3. Analysis of variance for predicting response surface equation

Table 6 gives the ANOVA for the second order response surface equation which shows that the second order response surface model fits well into the observed data. It also shows that both first and second order terms are significant at 5 and 1% level of significance.

6. Conclusions

Sintered high speed steel (HSS) components have been manufactured by powder metallurgy (PM) process. Water-atomized and vacuum-annealed powders of T15 HSS along with other ingredients like Zn-stearate (2%) and alumina (Al_2O_3) were used to produce the components. The percentage of alumina (x_1), sintering temperature (x_2) and sintering time (x_3) were considered as the controllable process parameters while the hardness of the sintered components was considered as the response variable. A 2^3 full factorial design of experiments (DOE) was used to collect experimental data to statistically analyze the effect of process parameters on the hardness of sintered HSS components. It has been observed that the percentage of alumina (x_1), sintering temperature (x_2) and also their interaction ($x_1 \times x_2$) affects the hardness very significantly while duration of sintering temperature (x_3) does not affects the hardness significantly. A second order response surface model (RSM) has been used to develop a predicting equation of hardness based on the data collected using a statistical design of experiments known as central composite design (CCD). The Fisher's F-ratio test and analysis of variance (ANOVA) show that the observed data fits well into the assumed second order RSM model.

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