

Application of Taguchi method in optimization of process parameters of ODS tungsten heavy alloys

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Abstract. In the present work, a design of experiment (DOE) technique using Taguchi method, has been applied to optimize the properties of ODS tungsten heavy alloys (WHAs). In this work Taguchi method involves nine experiments groups for four processing parameters (compaction pressure, sintering temperature, binding material type, and oxide type) with three levels was implemented. The signal-to-noise (S/N) ratio and analysis of variance (ANOVA) were employed to obtain the optimal process parameter levels and to analyze the effect of these parameters on density, electrical conductivity, hardness and compressive strength values. The results showed that all the chosen factors have significant effects on all properties of ODS tungsten heavy alloys samples. The density, electrical conductivity and hardness increases with the increase in sintering temperature. The analysis of the verification experiments for the physical properties (density and Electrical conductivity) has shown that Taguchi parameter design can successfully verify the optimal parameters, where the difference between the predicted and the verified values of relative density and electrical conductivity is about 1.01% and 1.15% respectively.

Keywords: oxide dispersion strengthened; tungsten heavy alloys; Taguchi method; S/N ratio; ANOVA; density; electrical conductivity

1. Introduction

Oxide dispersion strengthened (ODS) tungsten heavy alloys have been under intensive development over the past four decades due to their excellent high-temperature creep strength and good oxidation resistance. Previous researches (Ryu and Hong 2003, Kim *et al.* 2009, Jing *et al.* 2008, Lee *et al.* 2007) on ODS tungsten heavy alloys showed that there are a number of factors that affect the properties of these alloys e.g., compaction pressure, sintering temperature, sintering

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time, binding material type, and oxide dispersed type. The optimization of these factors has significant effect to improve the properties and performance of ODS tungsten heavy alloys.

Taguchi experimental design is a well-known technique that provides a systematic and efficient methodology for process optimization. Taguchi philosophy is to design quality into the product rather than to inspect for it after its production. The quality of the product should be considered at the beginning; i.e., during the design stage of the product development and should continue through the production process (off-line quality) (Bhutt 2003). Taguchi method applies simple tools such as signal to noise ratio (S/N) and analysis of variance (ANOVA) to determine optimal conditions and effect of each factors on main properties (Akhgar *et al.* 2012). However, in Taguchi method, S/N ratio is a measure of quality characteristics and deviation from the desired value. The term signal represents the desirable value (mean) and the noise represents the undesirable value (standard deviation from mean) for the output characteristic (Raghunath 2007). The heart of the Taguchi philosophy is the 'quality loss function'. The quality loss function is a function that is defined in terms of the deviation of a design parameter from an ideal or target value (nominal value). The value of the overall loss function is further transformed into a signal-to-noise (S/N) ratio. The S/N ratios play a great role in performing the ANOVA of the conducted experimental studies (Roy 2001, Rohit 2010). The main disadvantage of Taguchi method is that the results obtained are only relative and do not exactly indicate what parameter has the highest effect on the performance characteristic value. Taguchi does not consider higher order (nonlinear effect) interactions between design parameters. Also, since orthogonal arrays do not test all variable combinations, this method should not be used with all relationships between all variables. Another limitation is that Taguchi methods is offline, and therefore inappropriate for a dynamically changing process such as simulation studies. Furthermore, since Taguchi methods deal with designing quality rather than correcting for poor quality, they are applied most effectively at early stages of process development (Zu *et al.* 1997, Berginc *et al.* 2006).

The response surface methodology (RSM) is one of the most widely used methods to solve the optimization problem in the manufacturing environments (Sadhana 2011). Response surface methodology (RSM) is a collection of mathematical and statistical techniques that are useful for the modelling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response.

Generally, Taguchi approach uses three major steps namely system design, parameter design and tolerance design for optimizing a process or product. System design focuses on determining the suitable working levels of design factors. It includes designing and testing systems based on the researcher's judgment of selected materials, parts and technology. It also involves innovation and knowledge from applicable fields of sciences and technology. Parameter design aims at determining the control factors and their levels that produce the best performance of the product/process under study. The optimal condition is selected so that the influence of uncontrollable factors (noise factors) causes minimum variation of system performance (i.e., to make the design robust). Tolerance design is a way to fine-tune the results of the parameter design by tightening the tolerance of factors with significant influence on the product (i.e., development of specification limits) (Bhutt 2003, Raghunath 2007, Kamaruddin *et al.* 2004).

Therefore, the present work aims to finding out the effect of processes parameters like compaction pressure, sintering temperature, binding material type and oxide type on physical and mechanical properties of ODS tungsten heavy alloys. Optimizing of ODS tungsten heavy alloys properties through design of experiments allows for direction improvement efforts to enhance a product's manufacturability, reliability, quality, and field performance. In the current work

Table 1 Factors and their levels selected for the experiments

Symbol	Parameters	Levels		
		1	2	3
A	Compaction pressure (MPa)	200	400	600
B	Sintering temp. (°C)	1300	1400	1500
C	Binding material type	Ni-Fe	Ni-Co	Ni-Fe-Co
D	Oxide type	Y ₂ O ₃	ZrO ₂	TiO ₂

Table 2 Experimental conditions according to selected orthogonal array

Exp. No.	Samples (wt.%)	Pressure (MPa)	Sintering temp. (°C)
1	91W-4.9 Ni-2.1 Fe-2Y ₂ O ₃	200	1300
2	91W-4.9 Ni-2.1 Co-2 ZrO ₂	200	1400
3	91W-4.9 Ni-1.05 Fe-1.05 Co-2TiO ₂	200	1500
4	91W-4.9 Ni-2.1 Co-2TiO ₂	400	1300
5	91W-4.9 Ni-1.05 Fe-1.05 Co-2Y ₂ O ₃	400	1400
6	91W-4.9 Ni-2.1 Fe-2ZrO ₂	400	1500
7	91W-4.9 Ni-1.05 Fe-1.05 Co-2ZrO ₂	600	1300
8	91W-4.9 Ni-2.1 Fe-2TiO ₂	600	1400
9	91W-4.9 Ni-2.1 Co-2Y ₂ O ₃	600	1500

experiments were planned by using Taguchi's L_9 orthogonal array.

2. Experimental designs

2.1 Selection of orthogonal array

In this investigation, an experimental matrix was designed according to Taguchi experimental design method to create robust design. First, the appropriate orthogonal array for the control parameters and their levels to fit a specific study is selected. In this investigation four parameters in three levels mean that the L_9 (3^4) orthogonal array of Taguchi design have to be considered. The used parameters and levels are presented in Table 1. The numbers 1, 2 and 3 represent the lowest, mid and highest levels, respectively.

Tungsten alloys consisting of 91 wt.% of W metal, 7wt.% of (Ni-Fe, Ni-Co or Ni-Fe-Co) metallic binder were reinforced with 2 wt.% of Y₂O₃, ZrO₂, or TiO₂ particles and were fabricated by powder metallurgy techniques according to conditions determined by Taguchi orthogonal array (see Table 2).

2.2 Analysis of experimental data

Experimental data were analyzed by using S/N ratio and ANOVA. Based on the results of the S/N ratio and ANOVA, optimal parameter settings for better response were obtained and verified

Table 3 Experimental data and sample statistics for density

Exp. No	Replication relative density %			Mean	Standard deviation	S/N Ratio
	R ₁	R ₂	R ₃			
1	88.11	87.50	88.32	87.97	0.42	38.88
2	93.50	91.33	91.33	92.05	1.25	39.27
3	96.60	95.69	96.05	96.11	0.45	39.65
4	95.58	97.31	96.00	96.29	0.90	39.67
5	92.14	91.44	91.46	91.68	0.39	39.24
6	93.05	95.82	94.64	94.50	1.39	39.50
7	90.20	91.36	90.44	90.66	0.61	39.14
8	92.55	92.31	92.31	92.39	0.13	39.31
9	90.37	88.00	87.60	88.65	1.49	38.95

Table 4 The average S/N response table for density

Level	A	B	C	D
1	39.27	39.24	39.24	39.03
2	39.47	39.28	39.30	39.31
3	39.14	39.37	39.35	39.55
Delta	0.33	0.13	0.11	0.52
Rank	2	3	4	1

experimentally. In general, there are three categories of the quality characteristic in the analysis of the S/N ratio, i.e., the lower-the-better (LB), the higher-the-better (HB), and the nominal-the-better. In this investigation the higher-the-better S/N ratio using Eq. (1) was used for density, electrical conductivity, hardness and compressive strength of ODS tungsten heavy alloys. Regardless of the category of the quality characteristic, a larger S/N ratio corresponds to a better quality characteristic. Therefore, the optimal level of the process parameters is the level with the highest S/N ratio (Janmanee and Muttamara 2011, Yildiz and Gur 2011).

$$\text{The higher, the better: } \frac{S}{N} = \eta = -10 \log \left(\frac{1}{n} \sum_{i=1}^n \frac{1}{y_i^2} \right) \quad (1)$$

Where y_i is the performance characteristic and n is the number of experiments.

3. Results and discussion

3.1 Physical properties analysis using Taguchi method

3.1.1 Sintered densities of ODS tungsten heavy alloys

In order to maximize the sintered density of ODS tungsten heavy alloys samples; the higher the better S/N ratio for each run was calculated according to Eq. (1) and was recorded in Table 3.

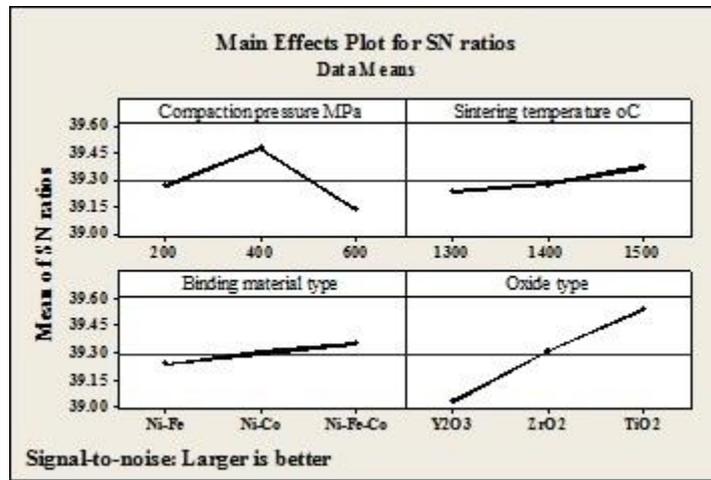


Fig. 1 Mean of S/N ratios for density of samples versus levels of input parameters

Table 5 ANOVA data for density of sintered parts

Factor	DOF	Sum of squares (SS)	Mean of squares (MS)	Variance ratio (F)	Percent contribution %	P value
A	2	58.56	29.28	35.13	25.87	0.00
B	2	10.03	5.01	6.02	4.43	0.01
C	2	6.52	3.26	3.91	2.88	0.03
D	2	136.20	68.10	81.70	60.18	0.00
Error	18	15.00	0.83		6.63	
Total	26	226.32			100	

The response table of the testing process for the four selected factors is presented in Table 4. The control factor with the strongest influence was determined by difference values. The higher the difference, the more influential the control factor becomes.

From the response table it is obvious that the strongest influence was of the oxide type D. These results confirm the previous research findings on the effect of adding oxides to tungsten heavy alloys (Kim *et al.* 2009, Jing *et al.* 2008, Veleva 2011, Veleva *et al.* 2009). These reports show that adding rare earth metals oxide particles to W leads to grain refinement and increases the relative density. This is because the dispersed oxides retard the grain coarsening during the liquid phase sintering.

The main effects plot for S/N ratios is shown in Fig. 1. The graph is constructed in order to understand the effects of parameters and their levels on the density. It is obvious that the control factor of compaction pressure (A) at level 2 (400 MPa) provided the best response. Similarly, the sintering temperature (B) at level 3 (1500°C), the binding material type (C) at level 3 (Ni-Fe-Co) and oxide type (D) at level 3 (TiO₂) provided the best response. Therefore, the optimal parameter combination level identified for the present investigation in the process is A₂B₃C₃D₃ for maximum sintered density.

ANOVA was used to investigate the statistical significance of the parameters at 95% confidence level and to determine the percentage of contribution of the parameters to the process

Table 6 Experimental data and sample statistics for electrical conductivity

Exp. No	Replication Electrical resistivity ($\mu\Omega\cdot\text{cm}$)			Electrical conductivity ($\mu\Omega^{-1}\cdot\text{cm}^{-1}$)			Mean	Standard deviation	S/N Ratio
	R ₁	R ₂	R ₃	R ₁	R ₂	R ₃			
	1	7.495	8.070	7.741	0.133	0.123			
2	6.220	5.830	6.350	0.160	0.171	0.157	0.162	0.0073	15.79
3	6.125	5.789	5.701	0.163	0.172	0.175	0.170	0.0062	15.40
4	7.170	7.359	6.991	0.139	0.135	0.143	0.139	0.0040	-17.14
5	6.072	5.710	6.468	0.164	0.175	0.154	0.164	0.0105	15.72
6	6.365	6.245	6.435	0.157	0.160	0.155	0.157	0.0025	16.06
7	8.664	8.489	8.650	0.115	0.117	0.115	0.115	0.0011	-18.73
8	6.576	6.426	6.466	0.152	0.155	0.154	0.153	0.0015	16.26
9	6.453	6.197	6.235	0.154	0.161	0.160	0.158	0.0037	-16.01

Table 7 The average S/N response table for electrical conductivity

Level	A	B	C	D
1	-16.35	-17.91	-16.73	-16.53
2	-16.31	-15.93	-16.32	-16.86
3	-17.01	-15.83	-16.62	-16.27
Delta	0.70	2.08	0.41	0.59
Rank	2	1	4	3

response. The significance of each parameter was tested using probability values (p-value). When the p-value in the ANOVA table is less than 0.05 (95% confidence level), it is considered that the parameters are statistically significant (Kasman 2013). According to the ANOVA results for densities given in Table 5, the p-values show that, all the parameters are statistically significant, implying that the parameters have significant impact on the density value because their p-values are less than 0.05. The results also show that the oxide type with 60.18% contribution and compaction pressure with 25.87% contribution have the highest influence on the density. These results are in good agreement with S/N ratio analysis.

Once the optimal level of the design parameters has been determined, the final step is to predict and verify the improvement of the quality characteristic using the optimal level of the design parameters. For this study $A_2B_3C_3D_3$ are the optimal levels of the processing parameters for gaining the maximum density. Hence, the expected average density at the optimal condition could be estimated by using Eq. (2) (Rohit 2010, Ji *et al.* 2001).

$$Density_{predicted} = T + (A_2 - T) + (B_3 - T) + (C_3 - T) + (D_3 - T) \quad (2)$$

Where T is the current grand average of performance. A_2 , B_3 , C_3 and D_3 are the average responses when each factor is at the highest density. The relative density predicted at optimal condition=98.22%.

After predicting of the optimal density, the verification experiment was performed in order to

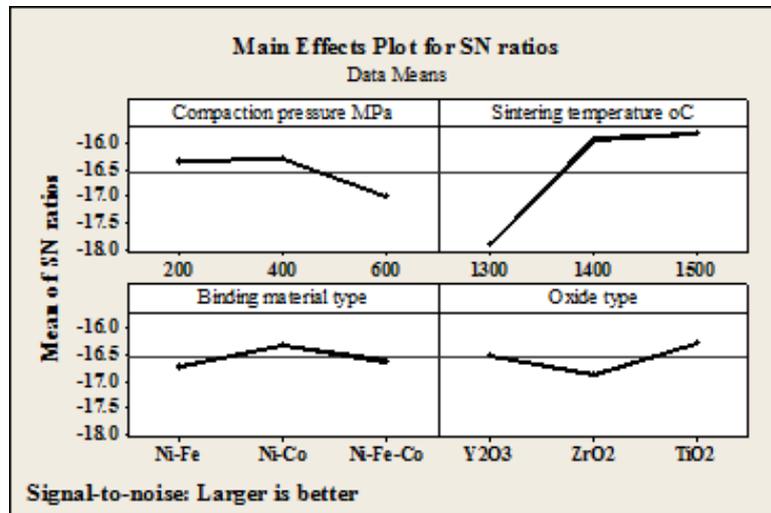


Fig. 2 Mean of S/N ratios for electrical conductivity of samples versus levels of input parameters

determine the difference between the predicted and measured values. The condition $A_2B_3C_3D_3$ of the optimal parameter combination was treated as verification run. Three specimens were prepared under the optimal parameter set up in the study. The mean relative density of the verified specimens was 97.23%. This result indicates that the selected control factor levels produced the best density. The difference between the predicted relative density and the verified relative density is about 1.01% which means that predicted and verified values are very close to each others.

3.1.2 Electrical conductivities of ODS tungsten heavy alloys

The electrical resistivity of investigated samples was measured and the electrical conductivities were calculated for each experiment shown in the orthogonal array. The S/N ratio (the bigger the better) was calculated in order to maximize electrical conductivity of ODS tungsten heavy alloys. The experimental values of the electrical resistivity and the corresponding electrical conductivity with its S/N ratio for each sample are reported in Table 6. The response table of the electrical conductivity is presented in Table 7.

It is clear from Table 7 that the factor B (sintering temperature) has the highest effect on the electrical conductivity followed by the compaction pressure (A).

The response graph for electrical conductivity is shown in Fig. 2. It is obvious that electrical conductivity is maximum at the 2nd level of parameter A, 3rd level of parameter B, 2nd level of parameter C and 3rd level of parameter D. Therefore, the best levels for maximum electrical conductivity are $A_2B_3C_2D_3$.

It is noticed from Fig. 2 that electrical conductivity increases with increasing sintering temperature. One reason for the higher electrical conductivity of the ODS tungsten heavy alloy is attributed to the lower porosity content at the higher sintering temperatures and also the presence of oxides that reduce the porosity content due to its affinity to inhibit oxidation. As the porosity content increases, the electrical resistivity increase and this leads to lower values of electrical conductivities (Ibrahim *et al.* 2009). Moreover, the electrical conductivity increases by increasing compaction pressure until 400 MPa then decrease at 600 MPa. This may be attributed to high porosity forming at higher compaction pressure.

Table 8 ANOVA for electrical conductivity of sintered parts

Factor	DOF	Sum of squares (SS)	Mean of squares (MS)	Variance ratio (F)	Percent contribution %	p-value
A	2	0.00073	0.00036	12.25	8.57	0.000
B	2	0.00670	0.00335	111.97	78.34	0.000
C	2	0.00021	0.00010	3.57	2.50	0.049
D	2	0.00036	0.00018	6.13	4.29	0.009
Error	18	0.00053	0.00002		6.30	
Total	26	0.00855			100	

Table 9 Experimental data and sample statistics for hardness

Exp. No	Replication Hardness (HV)			Mean	Standard deviation	S/N Ratio
	R ₁	R ₂	R ₃			
1	279.40	225.00	222.40	242.26	32.18	47.54
2	345.00	316.20	322.20	327.80	15.19	50.29
3	348.60	342.60	354.20	348.46	5.80	50.84
4	373.40	397.20	386.40	385.66	11.91	51.71
5	355.60	339.80	355.60	350.33	9.12	50.88
6	347.20	338.40	338.60	341.40	5.02	50.66
7	291.60	290.80	291.00	291.13	0.41	49.28
8	345.80	342.00	335.80	341.20	5.04	50.65
9	379.60	359.20	361.40	366.73	11.19	51.27

Table 10 The average S/N response table for hardness

Level	A	B	C	D
1	49.56	49.51	49.62	49.90
2	51.09	50.61	51.10	50.08
3	50.41	50.93	50.34	51.07
Delta	1.53	1.42	1.48	1.17
Rank	1	3	2	4

Table 8 shows the ANOVA results for the electrical conductivity. It can be found that the sintering temperature with 78.34% contribution has the highest influence on the electrical conductivity while other factors have comparatively smaller effects. The p-values show that, all parameters have significant effect on electrical conductivity at 95% confidence level.

The results of this investigation show that the optimal levels of the processing parameters to maximize electrical conductivity were $A_2B_3C_2D_3$. Hence, the expected average of electrical conductivity at the optimum condition could be estimated by using Eq. (2) with the same way as the density. The predicted electrical conductivity value at the optimum conditions was $0.173 \mu\Omega^{-1} \cdot \text{cm}^{-1}$.

The verification experiment was conducted at the optimized factor levels $A_2B_3C_2D_3$. The value

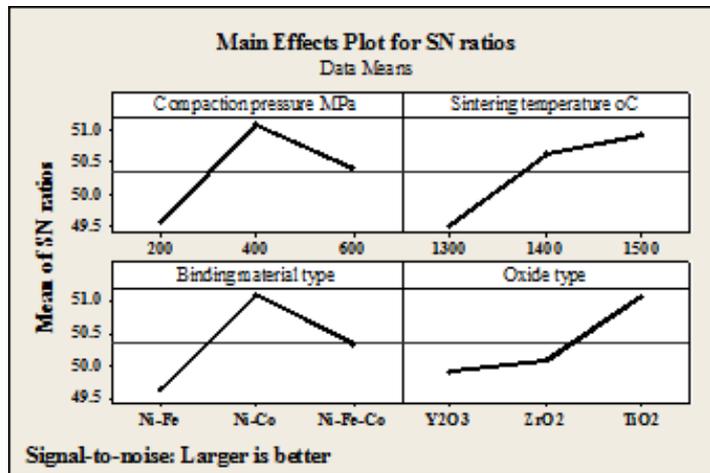


Fig. 3 Mean of S/N ratios for hardness of samples versus levels of input parameters

of electrical conductivity was calculated from the verified electrical resistivity. The electrical conductivity value for verification experiment is measured to be $0.171 \mu\Omega^{-1} \cdot \text{cm}^{-1}$, which is in confidence interval of the predicted electrical conductivity ($0.173 \mu\Omega^{-1} \cdot \text{cm}^{-1}$) where there is 1.15% difference between predicted and verified electrical conductivity.

3.2 Mechanical properties analysis using Taguchi method

3.2.1 Hardness of ODS tungsten heavy alloys

Experimental measured values and S/N ratios for hardness of ODS tungsten heavy alloys samples are reported in Table 9. The response table of the hardness analysis using Taguchi approach is presented in Table 10.

From the results in Table 10 it is obvious that the factors that have the strongest influence on hardness were compaction pressure (A), binding material type (C), sintering temperature (B), and oxide type (D) respectively.

The S/N response graph for hardness is shown in Fig. 3. According to this graph it is obvious that the optimal hardness of ODS tungsten heavy alloys could be attained at the combined settings of A_2 , B_3 , C_2 and D_3 .

Fig. 3 shows that the hardness of ODS tungsten heavy alloys increases with increasing compaction pressure until 400 MPa then decrease at 600 MPa. Hardness is affected by the level of porosity where, the hardness increases as the porosity content decreases. This is because high pressures prevent the evaporated lubricants (paraffin wax) and/or moisture from escaping during sintering and lead to form porosity as mentioned before (Ibrahim *et al.* 2009).

ANOVA results for the hardness are shown in Table 11. As it is shown in the table, the compaction pressure with 26.73% contribution and binder material type with 25.78% contribution have the most influence on the hardness while sintering temperature (21.44%) and oxide type (18.84%) have comparatively smaller effect on the hardness of the samples. The p-values in Table 11 show that, all parameters have significant effects on hardness at 95% confidence level.

According to S/N and ANOVA the optimal levels of the processing parameters to maximize hardness was determined. For this investigation $A_2B_3C_2D_3$ was the best combination of factors

Table 12 Experimental data and sample statistics for compressive strength

Exp. No	Replication Compressive strength at $\epsilon=0.5$			Mean	Standard deviation	S/N Ratio
	R ₁	R ₂	R ₃			
1	855.00	858.00	823.00	845.33	19.39	58.53
2	2020.60	2090.00	2036.00	2048.87	36.44	66.22
3	2094.00	2019.36	2060.00	2057.79	37.36	66.26
4	1990.00	2000.38	2025.60	2005.33	18.30	66.04
5	2017.20	2062.04	2093.20	2057.48	38.20	66.26
6	2080.30	2049.78	2027.38	2052.49	26.56	66.24
7	1770.60	1790.90	1755.78	1772.43	17.63	64.97
8	2009.45	2045.76	2034.00	2029.74	18.52	66.14
9	1980.90	1995.34	2010.50	1995.58	14.80	66.00

Table 13 The average S/N response table for compressive strength

Level	A	B	C	D
1	63.68	63.18	63.64	63.60
2	66.18	66.21	66.09	65.81
3	65.71	66.17	65.83	66.15
Delta	2.50	3.03	2.45	2.55
Rank	3	1	4	2

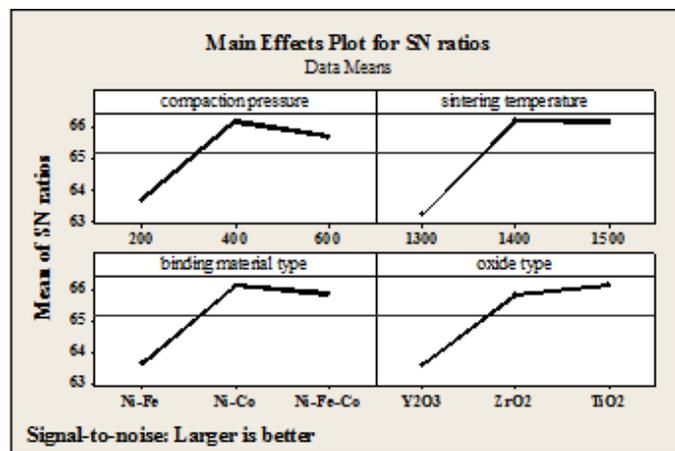


Fig. 4 Mean of S/N ratios for compressive strength of samples versus levels of input parameters

levels in order to optimized hardness of ODS tungsten heavy alloys. Hence, the predicted hardness value at optimal condition was 431.51 HV.

A Verification experiment was conducted according to the optimized factors levels $A_2B_3C_2D_3$. Three specimens were prepared under the optimal conditions. The hardness is measured to be 410.67 HV, which is in confidence interval of the predicted hardness (431.51 HV) where the difference between the predicted and verified was about 4.8%.

Table 14 ANOVA for compression strength of sintered parts

Factor	DOF	Sum of squares (SS)	Mean of squares (MS)	Variance ratio (F)	Percent contribution %	p-value
A	2	723142	361571	502.60	19.14	0.000
B	2	1496223	748112	1039.91	39.62	0.000
C	2	735834	367917	511.42	19.48	0.000
D	2	808699	404350	562.06	21.41	0.000
Error	18	12949	719		0.34	
Total	26	3776847			100	

3.2.2 Compressive strength of ODS tungsten heavy alloys

The results of measured values and the corresponding S/N ratios for compressive strength at $\epsilon=0.5$ of ODS tungsten heavy alloys samples are given in Table 12 and the response table is presented in Table 13.

It is evident from the response table that the sintering temperature has the largest significant effect on compressive strength then oxide type, compaction pressure and binding material type respectively.

Fig. 4 shows the response graph of compressive strength. It is obvious that compressive strength of the samples has optimal value at 400 MPa (A_2), 1400°C (B_2), Ni-Co (C_2) and TiO₂ (D_3).

ANOVA results for the compressive strength (Table 14) indicate that sintering temperature with 39.62% contribution is the most effective parameter on the compressive strength of ODS tungsten heavy alloys samples.

According to p-values, all parameters have significant effects on compressive strength at 95% confidence level. These results are in agreement with the S/N ratio results.

The optimal levels of the processing parameters to maximize compressive strength of ODS tungsten heavy alloys was determine from response graph as $A_2B_2C_2D_3$. Hence, the expected average of compressive strength at the optimum condition was calculated as 2509.66 MPa. The verification experiment was conducted according to the optimized factors levels $A_2B_2C_2D_3$. The compressive strength is measured to be 2833 MPa. The difference between the predicted and verified value of compressive strength was 12.8%, this value is very large relative to the corresponding differences for physical properties.

4. Conclusions

The main conclusions drawn from this investigation are summarized as follows:

1. Statistical results using ANOVA (at a 95% confidence level) show that the oxide type (D), compaction pressure (A), sintering temperature (B) and binding material type (C) affect the sintered density of ODS tungsten heavy alloys by 60.18%, 25.87%, 4.43% and 2.88% respectively.
2. The analysis of the verification experiments for the physical properties (density and Electrical conductivity) has shown that Taguchi parameter design can successfully verify the optimal parameters, where the difference between the predicted and the verified values of relative density and electrical conductivity is about 1.01% and 1.15% respectively.
3. ANOVA results for all experiments show that all the selected parameters for this investigation have significant effect on the mechanical and physical properties of ODS tungsten

heavy alloys.

4. From the detected results of this investigation no doubt that implementation of Taguchi technique can be successfully applied through designing smallest experiments with selected factors in order to improve the performance of ODS tungsten heavy alloys.

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