

Surface Integrity of Ni-Rich NiTi Shape Memory Alloy at Optimized Level of Wire Electric Discharge Machining **Parameters**

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The concern of this experimental work is to study the surface integrity aspects such as surface morphology, three-dimensional surface topography, recast layer, phase analysis, and micro-hardness for $Ni_{55.95}Ti_{44.05}$ shape memory alloy at the optimized level of wire electric discharge machining parameters. A mathematical model was developed for surface roughness and material removal rate considering servo voltage, pulse on time, wire tension, wire feed rate, and pulse off time using response surface methodology technique. In order to obtain the optimized parameters, multi-objective optimization technique grey relation analysis was utilized. The adequacy of the developed model was also checked by analysis of variance. At optimal parameters setting, i.e., pulse on time 123 µs, pulse off time 58 µs, servo voltage 50 V, wire tension 3 N, and .
wire feed rate 5 m/min, maximum material removal rate (8.223 mm³/min) and minimum surface roughness (1.93 lm) were achieved. Surface characteristics of machined surface divulge the presence of discharge craters, debris, molten droplets, micro-voids, spherical nodules, and cracks. A recast layer of thickness 19 lm with approximately 21% of foreign elements was deposited on the machined surface at optimized parameters, whereas the micro-hardness of the outer machined surface was found to be increased approximately 1.98 times as compared to micro-hardness of bulk material. X-ray diffraction analysis shows the presence of the following compounds on the machined surface NiTi, Ni_4Ti_3 , Ti_4O_3 , Cu_5Zn_8 , $Ni(TiO_3)$, and NiZn.

Keywords optimization, RSM, shape memory alloy, surface integrity aspects, WEDM

1. Introduction

A wide spectrum of NiTi SMAs' unique properties stimulates the interest of its usage in many sectors such as aerospace, biomedical, robotics, and other important domains because of their exceptional properties (Ref [1-5](#page-11-0)). In the past few decades, NiTi SMAs undergo considerable modifications in their phenomenal properties like shape memory effect, high wear and corrosion resistant, superelasticity and high specific strength. The same properties that impart uniqueness are also responsible for poor machinability in NiTi SMAs (Ref [6-8\)](#page-11-0). A great proportion of SMA product market is occupied by Ni-rich SMAs (Ref [9\)](#page-11-0). Owing to the poor machinability with conventional methods, it is difficult to maintain simultaneously low surface roughness, high MRR, and high dimensional accuracy (Ref [10](#page-11-0), [11](#page-11-0)). Therefore, these issues must be dealt with extra care; to control these responses, the advanced machining processes must be exploited for effective and profitable machining of NiTi SMAs. However, unconventional machining processes, namely laser beam machining, chemical and elec-

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List of Symbols					
T_{ON}	Pulse on time (μs)				
$T_{\rm OFF}$	Pulse off time (μs)				
$W_{\rm g}$	Spark gap				
Wire diameter d					
	Abbreviations				
WEDM	Wire electric discharge machining				
SMA	Shape memory alloy				
SV.	Servo voltage (V)				
WT.	Wire tension (N)				
WF	Wire feed rate (m/min)				
MRR	Material removal rate (mm ³ /min)				
$R_{\rm a}$	Surface roughness (μm)				
RSM	Response surface methodology				
CCD	Central composite design				
SEM	Scanning electron microscope				
EDS	Energy-dispersive spectroscopy				
XRD	x-ray diffraction				
GRA	Grey relation analysis				
GRG	Grey relation coefficient				
GRC	Grey relation grade				
ANOVA	Analysis of variance				
SS	Sum of square				
MS	Mean square				
DOF	Degree of freedom				
СI	Confidence interval				

trochemical machining, abrasive waterjet machining, and electric discharge machining, have their own limitations like burr formation, low surface finish, heat-affected zone, and surface reactions. Among all these advanced machining processes, WEDM has comparatively better suitability for cutting hard and conductive alloys which are difficult to cut by conventional machining processes (Ref [12,](#page-11-0) [13](#page-11-0)). In WEDM process, a thin conductive traveling wire is used as electrode and material is removed by a series of rapidly repeated sparks between the workpiece and wire, separated by a dielectric fluid and subjected to electric voltage (Ref [14-16](#page-11-0)). It can machine any electrically conductive material regardless of the hardness and complex geometries (Ref [17](#page-11-0)).

Few researchers have attempted the machining of NiTi SMAs by WEDM. Bisaria and Shandilya (Ref [3](#page-11-0), [5](#page-11-0)) performed a parametric study for Ni-rich NiTi SMA during WEDM using one factor at a time approach. It was found that surface integrity aspects (recast layer, surface morphology, micro-hardness, phase analysis, etc.) of the machined surface were predominantly influenced by pulse on time, spark gap voltage, and pulse off time, whereas wire feed rate and wire tension were related to the accuracy, precision, and economics of machining. Hsieh et al. (Ref [12](#page-11-0)) examined the machining characteristics of NiTi-based ternary SMA in WEDM and concluded that longer pulse duration degraded the surface quality and increased the recast layer thickness. Manjaiah et al. (Ref [18\)](#page-11-0) have explored the machinability of $Ti_{50}Ni_{40}Cu_{10}$ and $Ti_{50}Ni_{30}Cu_{20}$ SMA in WEDM using full factorial design (FFD) approach and concluded that MRR and surface roughness increased with an increase in pulse on time and the decrease in pulse off time and servo voltage. New phases were detected on the machined surface because of the chemical reaction among wire electrode, dielectric, and machined surface at elevated temperature. Liu et al. (Ref [19](#page-11-0)) examined the properties, composition, and crystallography of white layer for SE508 Nitinol (50.8 at.% Ni-49.2 at.% Ti) in WEDM. A crystalline-structured white layer formed on the machined surface comprises the small number of foreign elements (copper and zinc) from brass wire electrode. The nano-hardness of the white layer was found to be increased compared to the bulk hardness of material due to oxide hardening. For multi-response optimization in WEDM process, researchers have effectively utilized GRA technique. Rajyalakshmi and Ramaiah (Ref [20](#page-11-0)) optimized the WEDM process parameters for Inconel 825 alloy using Taguchi and GRA. At the optimal set of parameters (using GRA), MRR was increased by 6.04%, whereas surface roughness and spark gap were reduced by 14.29 and 15.384%, respectively, which endorsed the capability of the optimization process. Saha and Mondal (Ref [21](#page-11-0)) experimentally studied the WEDM process for nanostructured hardfacing material and developed a mathematical model using RSM. Confirmatory experiments validating the Taguchi coupled with principal component analysis (PCA) enhanced the machining performance (MRR and surface roughness) and optimized WEDM process parameters. Majumder et al. (Ref [22](#page-11-0)) developed RSM model for cutting time and surface roughness of Inconel 800 in WEDM process and optimized the parameters using GRA technique. It was concluded that the predicted values by the developed model were closed to experimental values.

After a detailed scrutiny of the literature, it has been concluded that the domain of previous research works was directed to the effect of WEDM parameters on performance characteristics of NiTi SMAs. Some attempt has been made

keeping in view of the optimization, but the study on surface integrity aspects after optimization for Ni-rich NiTi SMA has been seldom reported which is novel and interesting aspects of this experimental study. The present study focuses on the modeling and multi-objective optimization of WEDM parameters for $Ni_{55.95}Ti_{44.05}$ SMA, considering MRR and R_a as response parameters and T_{ON} , T_{OFF} , SV, WF, and WT as variable parameters. CCD of RSM technique has been adopted for developing a mathematical model for MRR and R_a . For studying surface integrity aspects, GRA approach was used to find out optimized process parameters at which maximum MRR and minimum R_a can be achieved. Phase analysis, microhardness, surface characteristics, and recast layer have been studied under surface integrity aspects by using XRD, Vickers micro-hardness tester, SEM, optical profilometer, and EDS technique, respectively.

2. Materials and Methodology

2.1 Material

For the experimental studies, a square plate of $Ni_{55.95}Ti_{44.05}$ $(55.95-wt.\%$ Ni and $44.05-wt.\%$ Ti) SMA with a density of 6.7 g/cm³ and dimensions 165 mm \times 165 mm \times 6 mm has been procured. The elemental composition (wt.%) of Ni_{55.95}Ti_{44.05} SMA was detected by EDS test conducted at 20 kV accelerating voltage. The phenomena of shape recovery ability by the application of stress and heat are associated with martensitic phase transformation between austenite and martensite phase. The austenite start, austenite finish, martensite start, and martensite finish temperatures were detected 304.6, 329.34, 283.48, and 258.65 K, respectively, by differential scanning calorimeter test (Ref [3,](#page-11-0) [5](#page-11-0)).

2.2 Design of Experiments

The range and levels of WEDM parameters have been decided on the basis of preliminary experiments by using one parameter at a time technique. WEDM parameters such as T_{ON} , SV, WT, T_{OFF} , and WF have been considered as variable to examine their effects on R_a and MRR for Ni_{55.95}Ti_{44.05} SMA. The range and levels of variable parameters are represented in Table [1](#page-2-0). A thin brass wire of diameter 0.25 mm as an electrode has been selected in this study. The pulse peak current, pressure of the dielectric fluid (deionized water), and dielectric conductivity were fixed at 12 A, 1 kg/cm², and \pm 20-24 μ s/m, respectively. A CCD technique of RSM has been employed to design the experiments. RSM is a collection of statistical and mathematical techniques in which desired response is affected by multi-variables. RSM can be utilized for modeling and analysis of engineering problems (Ref [23\)](#page-11-0). During this process, the influence of all the process parameters on responses is collectively analyzed, instead of one factor at a time analysis.

The relation between response z and *i* variable factors y_1, y_2 , y_3, y_4, \ldots, y_i is represented according to Eq 1.

$$
z = \Phi(y_1, y_2, y_3 \ldots y_i) + \zeta \tag{Eq 1}
$$

where Φ is response function and ζ represents noise or error. Let us consider $f(z)$ is desirable response function and x_i , x_j , $\dots x_n$ are the independent variable parameters. The quadratic model for $f(z)$ can be written as follows:

$$
f(z) = \alpha_o + \sum_{i=1}^n \alpha_i x_i + \sum_{i=1}^n \alpha_{ii} x_i^2 + \sum_{i < j} \alpha_{ij} x_i x_j \tag{Eq 2}
$$

where α_o , α_i , α_j , and α_{ij} are intercept term, linear coefficient, quadratic coefficient, and interaction term, respectively. For this study T_{ON} , T_{OFF} , SV, WF, and WT are variable parameters and MRR and R_a are desirable response parameters.

CCD is a well-known technique for designing five-factor and five-level type problems owing to the flexibility and minimum error (Ref [23\)](#page-11-0). For constructing second-order model in CCD, each parameter is varied at five levels $(-2, -1, 0, 1,$ and 2). According to central composite rotatable design, with five process parameters at a full fraction, a total of 50 experiments have been performed for modeling and analysis as per given in Eq 3.

No. experimental runs
$$
= 2^{f} + 2 \times f + N_c
$$

= $2^{5} + 2 \times 5 + 8 = 50$ (Eq 3)

where f is factors and N_c is center points. This five-factor and five-level design includes $32(2^5)$ full factorial points, 10 axial points ($\alpha = \pm 2$), and 8 center points (zero levels). The design matrix for experiments and responses is shown in Table [2.](#page-3-0)

2.3 Experimental Procedure

The machining was conducted on computer numerical control, four axes, Ultracut-843, WEDM. The effect of WEDM process parameters, namely, T_{ON} , T_{OFF} , SV, WT, and WF, on MRR and R_a has been studied. The specimens with the dimension of 10 mm \times 10 mm \times 6 mm were machined. The digital stopwatch was used for calculating machining time. Equation 4 was utilized to calculate MRR (Ref [24\)](#page-11-0).

MRR ðmm3 =min) ¼ cutting speed ðmm/min) thickness of sample ðmm) width of cut ðmm) ðEq 4Þ

where thickness of sample is 6 mm; width of cut = $2W_g + d$; W_g is spark gap (0.02 mm); and d is wire diameter (0.25 mm).

 R_a (average surface roughness) of the machined surface has been measured by ''Mitutoyo SJ-410'' surface roughness tester. The surface roughness for the individual sample was recorded at five distinct points, and the average was considered as a response parameter. The stylus speed and cutoff length were 0.1 mm/s and 0.8 mm, respectively, used to evaluate the length of 4 mm. PANalytical X'PERT x-ray analyzer was utilized to analyze the phase change of machined surface using $Cu-K\alpha$ radiation at room temperature. The power was $45 \text{ kV} \times 40$ mA, and the scanning rate of 2θ (5°-100°) was 3° min⁻¹. JEOL JSM-6010LA SEM attached with EDS facility was used for the morphological analysis of machined surface. The thickness of the recast layer was measured by the analysis of a crosssectional SEM image of the machined surface with the help of ImageJ software by using Eq 5 (Ref. [7\)](#page-11-0).

Recast layer thickness =
$$
\frac{\text{Area of recast layer } (\mu m^2)}{\text{Length of recast layer } (\mu m)}
$$

\n(Eq 5)

Bruker contour GTK optical profiler was exploited for analyzing three-dimensional surface topography. For the analysis of three-dimensional surface topography, the sample area was $240 \times 180 \text{ }\mu\text{m}^2$. The three-dimensional surface topography can be also utilized for the measurement of the height of the protrusion and the depth of depression on the machined surface.

CSM micro-hardness tester having square-shaped pyramidal indenter of the diamond at the load of 250 mN and dwell time of 15 s has been utilized to measure micro-hardness. The micro-hardness was measured by Eq 6 (Ref [5](#page-11-0)).

$$
\text{Vicker hardness (Hv)} = \frac{2P\sin\frac{136^{\circ}}{2}}{D^2} \tag{Eq 6}
$$

where P is applied load (N) and D (mm) is the average of two diagonal indenters having an angle of 136°.

3. Results and Discussion

3.1 ANOVA for Material Removal Rate

The experimental results (given in Table [2](#page-3-0)) were analyzed by using Design Expert 9.0.5 software. ANOVA results for MRR are summarized in Table [3](#page-4-0). As per obtained fit summary from the analysis, it can be noticed that the quadratic model developed for MRR is statistically significant. In order to remove nonsignificant terms in the developed model, the backward elimination with a 95% CI is used. The F value of the model is 52.42 which validates that the developed model is significant. There is only 0.01% possibility that an F value this large could occur due to noise. The probability value (Prob > F or p value) $\langle 0.05 \rangle$ designates the significance of model terms. As per ANOVA results, for MRR, the parameters T_{ON} , T_{OFF} , SV, square of T_{ON} , square of T_{OFF} , square of SV and interaction of SV and T_{OFF} were the significant factors, whereas WF and WT were insignificant. The F value of lack of fit is 2.54 which infers that there is a 10.39% possibility that an F value of lack of fit'' of this large could occur due to noise. Determination coefficient (R^2) value is 0.9658 which indicates

that this developed model is capable to explain 96.58% variation and rest 3.42% variations may not be explained. The predicted R^2 (0.8832) is in realistic agreement with the adjusted- R^2 (0.9423) since the difference is \lt 0.2. Adequate precision measures the ratio of signal to noise (i.e., ratio of significant factors to nonsignificant factors); the value should be greater than 4 for a model to be adequate. The value of adequate precision is 25.60 (i.e., large signal-to-noise ratio) which also validates that the developed model is significant. Square of T_{ON} has the highest percentage contribution followed by T_{OFF} , T_{ON} , and SV.

The mathematical relationship between MRR and variable parameters in coded form is given in Eq [7](#page-4-0). The equation in coded form can be used to predict the value of MRR at a given level of variable parameters. The high levels of the parameters are assigned $+1$ code, and the low-level factors are coded as - 1. This coded equation is useful for finding the relative influence of the factors by comparing the coefficients of factors. Equation [8](#page-4-0) depicts the mathematical relationship in actual factors which can also be used to make predictions of MRR at a given level of each factor; however, the levels should be specified in the original units for each factor. This developed

Table 3 ANOVA table for material removal rate

Source	SS	DOF	MS	F value	p value Prob $> F$	Remarks	Percentage contribution
Model	52.42	20	2.62	40.99	< 0.0001	Significant	\cdots
A	7.20		7.20	112.60	< 0.0001	Significant	15.60
B	8.15		8.15	127.39	< 0.0001	Significant	17.66
\mathcal{C}	3.62		3.62	56.54	< 0.0001	Significant	7.84
BC	4.43		4.43	69.28	< 0.0001	Significant	9.59
A^2	19.44		19.44	304.02	< 0.0001	Significant	42.12
B ²	1.18		1.18	18.43	0.0002	Significant	2.55
C^2	0.28		0.28	4.32	0.0467	Significant	0.61
Residual	1.85	29	0.064				
Lack of fit	1.65	22	0.075	2.54	0.1039	Not significant	
Pure error	0.21	7	0.029				$R^2 = 0.9658$
Corrected total	46.15	49					Adjusted- R^2 = 0.9423 Predicted $R^2 = 0.8832$ Adequate precision = 25.60

equation is not sufficient to identify the relative influence of individual factor since the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

Figure $1(a)$ $1(a)$ and (b) shows the variation of the normal probability of residuals and the actual experimental value and predicted value for MRR. The experimental values are close to the predicted values with 95% CI. The residuals fall on the straight line in Fig. [1\(](#page-5-0)a), showing the normal distribution of error.

The final equation for MRR in terms of coded factors:

$$
\text{MRR (mm}^3/\text{min}) = 4.7151 + 0.4243 \times A - 0.4513 \times B - 0.3007 \times C - 0.3721 \times B \times C + 0.7795 \times A^2 + 0.1919 \times B^2 + 0.09289 \times C^2 \tag{Eq 7}
$$

The final equation for MRR in terms of actual factors:

$$
MRR (mm3/min) = 2821 - 46.0997 \times T_{ON} - 2.9188
$$

\times T_{OFF} + 2.2603 \times SV - 0.0372
\times T_{OFF} \times SV + 0.1949 \times T_{ON}^2
\n+ 0.048 \times T_{OFF}^2 + 0.0037 \times SV^2
\n(Eq 8)

3.2 Effect of WEDM Parameters on Material Removal Rate

Figure $1(c)$ $1(c)$, (d) , (e) , and (f) represents the effect of significant parameters such as T_{ON} , T_{OFF} , SV, and interaction of SV and T_{OFF} on MRR. As can be seen in Fig. [1\(](#page-5-0)c), MRR shows the increasing trend with T_{ON} . This is due to the fact that at a higher T_{ON} , the spark energy significantly increases which generates more heat to melt the material. In WEDM process, the energy needed to remove the material is achieved from the spark energy. So, the required thermal energy depends on the level of spark energy per pulse which finally converted into heat. The discharge energy is given by Eq 9 (Ref [5](#page-11-0)):

Discharge energy = Discharge voltage \times Discharge current \times Discharge duration

 $(Eq 9)$

As discharge voltage is almost constant, discharge duration and discharge current mainly influence the heating capability of individual spark. In case of zero ignition delay, the discharge duration becomes equal to pulse duration (i.e., T_{ON}). Discharge energy is directly influenced by T_{ON} . So, at higher T_{ON} , MRR increases due to substantial increase in the discharge energy.

Figure [1](#page-5-0)(d) shows the variation of MRR with T_{OFF} . The increase in MRR with the decrease in T_{OFF} can be observed from experimental results. It was observed that spark's energy and intensity of spark decreased at higher T_{OFF} which results in less removal of material from the workpiece, which finally led to lower MRR (Ref [3,](#page-11-0) [5\)](#page-11-0). Simultaneously, with the increase in T_{OFF} , sparks frequency decreases which also reduced the MRR. The spark frequency can be given as Eq 10 (Ref [5\)](#page-11-0):

Sparks frequency (kHz) $=$ $\frac{10^3}{T_{\text{ON}} + T_{\text{OFF}}}$ $(Eq 10)$

Figure [1](#page-5-0)(e) illustrates the effect of SV on MRR. The increase in SV has the inverse effect on MRR. This is due to the fact that the spark gap is widened at higher SV, which led to decrease sparking frequency (as given in Eq 10) and hence MRR finally decreased. Figure [1\(](#page-5-0)f) displays the 3D response plot for MRR in terms of SV and T_{OFF} . It was noticed from the response plot that MRR is decreased by increasing T_{OFF} from 54 to 58 µs and parallel increasing SV from 40 to 50 V. Similar effect of T_{ON} , T_{OFF} , and SV on MRR was also supported by Narendranath el al. (Ref [25\)](#page-11-0) and Manjaiah et al. (Ref [26\)](#page-11-0) findings for $Ti_{50}Ni_{42.4}Cu_{7.6}$ SMA and equiatomic NiTi SMA, respectively, in WEDM.

3.3 ANOVA for Surface Roughness

ANOVA results for R_a are tabularized in Table [4;](#page-5-0) the F value of the model is 35.50 which suggests the significance of the developed model. The parameters T_{ON} , T_{OFF} , SV, interaction of T_{ON} and SV, and square of T_{ON} are the significant model terms having a p value < 0.05 at 95% CI. It is observed that T_{OFF} has the highest percentage contribution followed by T_{ON} and SV for R_a . The probability value (p value) for lack of fit is 0.4658 infers that the lack of fit is not significant relative to the pure error. The R^2 for the model is 0.9607 shows the closeness of predicted values and actual experimental values. The predicted

Fig. 1 Residual graphs for MRR (a) normal probability vs. residuals, (b) actual experimental value vs. predicted value and effect of variable parameters, (c) TON, (d) T_{OFF} , (e) SV, (f) interaction of SV and T_{OFF} on MRR

Table 4 ANOVA table for surface roughness

Source	SS	DOF	МS	<i>F</i> value	p value Prob $>$ F	Remarks	Percentage contribution
Model	1.76	20	0.088	35.50	< 0.0001	Significant	.
A	0.43		0.43	173.80	< 0.0001	Significant	24.33
B	0.92		0.92	369.37	< 0.0001	Significant	52.06
\mathcal{C}	0.15		0.15	60.57	< 0.0001	Significant	8.49
	0.13		0.13	50.96	< 0.0001	Significant	7.36
AC A^2	0.065		0.065	26.23	< 0.0001	Significant	3.68
Residual	0.072	29	0.0024				
Lack of fit	0.056	22	0.0025	1.13	0.4658	Not significant	
Pure error	0.016	7	0.0022				$R^2 = 0.9607$
Corrected total	1.767	49					Adjusted- $R^2 = 0.9337$ Predicted $R^2 = 0.8749$ Adequate precision = 24.605

 R^2 (0.8749) is in realistic agreement with the adjusted R^2 (0.9337). For R_a , adequate precision is 24.605 which designates the adequate model discrimination. The developed mathemat-

ical equation for surface roughness in coded parameters and actual parameters is given in Eq [11](#page-6-0) and [12](#page-6-0), respectively. The normal probability of residual graph and the actual experimental value and predicted value graph for R_a are given in Fig. 2(a) and (b), respectively. The predicted values and experimental values at 95% CI are close as shown in Fig. 2(b).

The final equation for R_a in terms of coded factors:

$$
R_{\text{a}} \text{ } (\mu \text{m}) = 2.07 + 0.1038 \times A - 0.1513 \times B - 0.0613
$$
\n
$$
\times C + 0.0628 \times AC - 0.0451 \times A^2 \text{ (Eq 11)}
$$

The final equation in terms of actual factors:

$$
R_{\rm a} \, (\mu \rm m) = -129.949 + 2.4248 \times T_{\rm ON} + 0.1909 \times T_{\rm OFF} - 0.7857 \times \rm SV + 0.0063 \times T_{\rm ON} \times \rm SV - 0.0113 \times T_{\rm ON}^2
$$
\n(Eq 12)

3.4 Effect of WEDM Parameters on Surface Roughness

Figure $2(c)$, (d), (e) and (f) represents the effect of significant parameters, namely T_{ON} , T_{OFF} , SV, and interaction of SV and T_{ON} on surface roughness. Figure 2(c) displays the effect of T_{ON} on R_{a} . R_{a} was found to increase with an increase in T_{ON} . In WEDM, at a higher value of T_{ON} , discharge energy

[given in Eq [9\]](#page-4-0) is increased. At higher discharge energy, more heat is available to remove the material and hence deeper and wider craters are formed at the machined surface. Finally, SR increases due to the formation of the deeper craters at the machined surface at higher T_{ON} .

The effect of T_{OFF} on R_a is shown in Fig. 2(d). R_a shows the reverse trend with T_{OFF} . R_{a} decreases with an increase in T_{OFF} . This is because of an increase in flushing time (time to wipe out the molten droplets on the machined surface) at higher T_{OFF} (Ref [24\]](#page-11-0). Simultaneously, as T_{OFF} increases, sparking frequency decreases, and sparking-ampere increases. So, it is suggested that low sparking frequency is used for rough cutting operation, whereas high sparking frequency is preferred for the finishing operation.

Similar to T_{OFF} , the increase in SV has the inverse effect on R_a . Figure 2(e) shows the decreasing trend of R_a with SV. This is due to the reason that at higher SV spark gap widened which led to decrease in sparking frequency and finally surface roughness decreases. Three-dimensional response plot for surface roughness in terms of SV and T_{ON} is shown in Fig. 2(f). From the response plot, it can be seen that R_a is increasing by increasing T_{ON} from 117 to 121 µs and parallel decreasing SV from 40 to 50 V. Sharma et al. (Ref [27](#page-11-0)) and

Fig. 2 Residual graphs for surface roughness a normal probability vs. residuals, **b** actual experimental value vs. predicted value and effect of variable parameters, c TON, d T_{OFF} , e SV, f interaction of SV and TON on surface roughness

Manjaiah et al. (Ref [28](#page-11-0)) also encountered with similar findings for R_a during WEDM of $Ni_{40}Ti_{60}$ SMA and NiTiCu SMA, respectively.

4. Multi-objective Optimization

4.1 Grey Relational Analysis (GRA) Technique

Among multi-response optimization technique, GRA technique can be utilized for solving the complex interrelationship. In GRA technique, multi-response optimization problem can be converted into single relational grade. GRA technique has been utilized to optimize MRR and R_a for $\text{Ni}_{55.95}\text{Ti}4_{4.05}$ SMA. In the multi-optimization using GRA, the following steps are involved (Ref [29](#page-11-0)):

Step 1 Data preprocessing (Normalization of experimental data).

In GRA, first experimental results data are linearly normalized in the range from 0 to 1 which is known as grey relational generation. The experimental data can be normalized by three different approaches according to objective, i.e., ''smaller the better," "larger the better" and "nominal the better." The expression for normalizing the data using smaller the better and larger the better type characteristics is given Eq 13 and 14:

For larger the better type objective (e.g., MRR)

$$
z_i * (k) = \frac{\left[z_i^0(k) - \min\{z_i^0(k)\}\right]}{\left[\max\{z_i^0(k)\} - \min\{z_i^0(k)\}\right]}
$$
\n(Eq 13)

For smaller the better type objective (e.g., R_a)

$$
z_i * (k) = \frac{\left[\max\{z_i^0(k)\} - z_i^0(k)\right]}{\left[\max\{z_i^0(k)\} - \min\{z_i^0(k)\}\right]}
$$
 (Eq 14)

where $z_i^0(k)$ and $z_i^*(k)$ are the original sequence and sequence after the data processing, respectively, whereas max $z_i^0(k)$ is the largest value of $z_i^0(k)$ and min $z_i^0(k)$ is the smallest value of $z_i^0(k)$.

Step 2 Grey relation coefficient (GRC)

GRC represents the correlation between actual and desired experimental data. GRC is calculated as per Eq 15:

$$
\beta_{0,i}(k) = \frac{[\Delta_{\min} + \zeta \Delta_{\max}]}{[\Delta_{\text{o}i}^*(k) + \zeta \Delta_{\max}]} 0 < \beta_{0,i}(k) \le 1
$$
\n(Eq 15)

where $\beta_{0,i}$ (k) is grey relational coefficient, ζ is identification coefficients, $\zeta \in [0, 1]$ and generally 0.5 is used. Δ_{oi} (k) is the difference of the absolute value $z_0^*(k)$ and $z_i^*(k)$, i.e., Δ_{oi} $(k) = |z_0^*(k) - z_i^*(k)|$, $\Delta_{\min} = \min \{ \min \Delta_{\text{oi}} (k) \}$ and $\Delta_{\max} =$ max (max Δ_{oi} (k))

Step 3 Grey relational grade (GRG)

GRG is used for overall evaluation of multi-response characteristics. The average of GRC value is considered as GRG. GRG is given in Eq 16.

$$
\Upsilon_i = \frac{1}{m} \sum_{k=1}^{m} \beta_{0,i}(k) \tag{Eq 16}
$$

where m is the number of process response and the higher value of GRG signifies the closeness of parameters to the optimum solution. The higher value of GRG signifies the closeness of the corresponding set of parameters to the optimal.

The normalized value, deviation, GRC, and GRG values for individual experiment runs are given in Table [5](#page-8-0). Experimental run 34 has the highest GRG (1) representing the best combination among 50 experimental runs. At experimental run 34, i.e., A (123 μs), B (56 μs), C (45 V), D (4 N), E (4 m/ min), the max MRR (8.554 mm³/min) and min R_a (2.12 µm) were achieved among 50 experiments. The mean response for overall GRG is given in Table [6.](#page-9-0) It was observed that T_{ON} , T_{OFF} and SV were the significant parameters having the highest delta (max GRG–min GRG), whereas WF and WT are insignificant parameters for the improvement in GRG. This finding also validates the results of ANOVA for MRR and R_a . From Table [6](#page-9-0), it can be observed that at $A_2B_1C_1D_{-1}E_1$ parameter setting, i.e., A (123 μ s), B (58 μ s), C (50 V), D (3 N), and E (5 m/min), respectively, max MRR and min R_a can be achieved. $A_2B_1C_1D_{-1}E_1$ parameter setting is overall optimized level of input parameters suggested by GRA technique.

4.2 Confirmatory Experiment

Table [7](#page-9-0) shows the optimized values obtained from confirmatory tests. The max MRR was 8.223 mm³/min and min R_a was 1.93 µm obtained experimentally at optimized levels of parameters, whereas the value predicted by RSM model (Eq [8,](#page-4-0) [12](#page-6-0)) for MRR and R_a was 7.842 mm³/min and 2.01 μ m, respectively. The percentage error between the predicted and experimental value for both MRR and R_a lies \lt 5% which endorses the accuracy of developed RSM model. With respect to initial setting $(A_{-1}B_{-1}C_{-1}D_{-1}E_{-1})$, an increment of 21.73% and decrement of 9.34% for MRR and R_a , respectively, were observed at optimal levels of parameters.

5. Surface Integrity Aspects

The processing and end use of the machined component are directly affected by surface characteristics of the machined part. During WEDM process, surface characteristics of the machined part are altered. In surface integrity aspects, the altered surface characteristics which affect the properties of workpiece material are measured (Ref [30\)](#page-12-0). Surface integrity may be defined as the enhanced condition of the surface manufactured by any machining process or other processes related to surface generation. Surface integrity includes not only the topological (geometrical) aspects of the machined surface but also the physical, chemical, metallurgical, mechanical and biological aspect (Ref [31\)](#page-12-0).

After getting the optimized parameters setting $(A_2B_1C_1D_{-1}E_1)$, the surface integrity aspects such as surface morphology, surface topography, recast layer thickness, phase analysis, and micro-hardness analysis have been carried out at optimized parameter setting.

5.1 Surface Characteristics

Figure $3(a)$ $3(a)$ and (b) illustrates the surface morphology and three-dimensional (3D) surface topography of the machined surface for $Ni_{55.95}Ti_{44.05} SMA$ at optimized parameters setting

Table 5 Grey relational coefficient and grey relational grade for MRR and R_a

Std. run	Normal (MRR)	Normal (R_a)	Deviation (MRR)	Deviation (R_a)	GRC (MRR)	GRC (R_a)	GRG	Rank
$\mathbf{1}$	0.587	0.262	0.413	0.738	0.548	0.404	0.476	29
$\sqrt{2}$	0.649	0.202	0.351	0.798	0.588	0.385	0.486	26
\mathfrak{Z}	0.485	0.607	0.515	0.393	0.492	0.560	0.526	16
$\overline{\mathcal{L}}$	0.629	0.583	0.371	0.417	0.574	0.545	0.560	9
5	0.556	0.524	0.444	0.476	0.530	0.512	0.521	19
6	0.649	0.155	0.351	0.845	0.587	0.372	0.480	27
$\sqrt{ }$	0.312	0.881	0.688	0.119	0.421	0.808	0.614	$\overline{4}$
$\,$ $\,$	0.307	0.512	0.693	0.488	0.419	0.506	0.463	32
9	0.558	0.298	0.442	0.702	0.531	0.416	0.473	30
$10\,$	0.640	0.131	0.360	0.869	0.581	0.365	0.473	31
11	0.478	0.607	0.522	0.393	0.489	0.560	0.525	18
12	0.624	0.571	0.376	0.429	0.571	0.538	0.555	12
13	0.610	0.595	0.390	0.405	0.562	0.553	0.557	10
14	0.724	0.143	0.276	0.857	0.645	0.368	0.507	22
15	0.126	0.857	0.874	0.143	0.364	0.778	0.571	8
16	0.226	0.440	0.774	0.560	0.392	0.472	0.432	38
17	0.479	0.262	0.521	0.738	0.490	0.404	0.447	35
18	0.789	0.143	0.211	0.857	0.703	0.368	0.536	13
19	0.482	0.607	0.518	0.393	0.491	0.560	0.526	17
$20\,$	0.711	0.583	0.289	0.417	0.634	0.545	0.589	7
21	0.448	0.643	0.552	0.357	0.475	0.583	0.529	14
22	0.716	0.155	0.284	0.845	0.638	0.372	0.505	23
23	0.224	1.000	0.776	0.000	0.392	1.000	0.696	$\boldsymbol{2}$
24	0.283	0.619	0.717	0.381	0.411	0.568	0.489	25
25	0.348	0.238	0.652	0.762	0.434	0.396	0.415	40
$26\,$	0.740	0.107	0.260	0.893	0.658	0.359	0.508	21
$27\,$	0.365	0.690	0.635	0.310	0.441	0.618	0.529	15
28	0.722	0.440	0.278	0.560	0.642	0.472	0.557	11
29	0.535	0.500	0.465	0.500	0.518	0.500	0.509	$20\,$
30	0.854	0.274	0.146	0.726	0.774	0.408	0.591	6
31	0.107	0.988	0.893	0.012	0.359	0.977	0.668	3
32	0.482	0.488	0.518	0.512	0.491	0.494	0.493	24
33	0.575	0.738	0.425	0.262	0.540	0.656	0.598	5
34	1.000	0.274	0.000	0.726	1.000	0.408	0.704	$\mathbf{1}$
35	0.453	0.000	0.547	1.000	0.478	0.333	0.405	42
36	0.044	0.679	0.956	0.321	0.343	0.609	0.476	28
37	0.315	0.167	0.685	0.833	0.422	0.375	0.399	45
38	0.000	0.405	1.000	0.595	0.333	0.457	0.395	47
39	0.350	0.429	0.650	0.571	0.435	0.467	0.451	34
40	0.325	0.429	0.675	0.571	0.426	0.467	0.446	36
41	0.358	0.357	0.642	0.643	0.438	0.438	0.438	37
42	0.364	0.417	0.636	0.583	0.440	0.462	0.451	33
43	0.098	0.429	0.902	0.571	0.357	0.467	0.412	41
44	0.183	0.274	0.817	0.726	0.380	0.408	0.394	48
45	0.141	0.440	0.859	0.560	0.368	0.472	0.420	39
46	0.108	0.321	0.892	0.679	0.359	0.424	0.392	49
47	0.065	0.333	0.935	0.667	0.348	0.429	0.388	50
48	0.134	0.321	0.866	0.679	0.366	0.424	0.395	46
49	0.151	0.333	0.849	0.667	0.371	0.429	0.400	44
50	0.170	0.345	0.830	0.655	0.376	0.433	0.405	43

 $(A_2B_1C_1D_{-1}E_1)$, respectively. From the SEM micrograph shown in Fig. [3](#page-9-0)(a), the presence of many pockmarks, spherical nodules, micro-voids, craters, drops of molten material, and micro-cracks on the machined surface can be distinctly observed. In WEDM process, the successive electrical sparks led to a transfer of intense heat into the machined surface result in the generation of discharge craters. The size of craters decides the quality of the machined surface (Ref [32](#page-12-0)). During T_{OFF} , some of the molten material was wiped out by the deionized water. However, the melted material remains left resolidified on the machined surface to form lumps of debris. Pockmarks are formed on the machined surface due to entrap gases escaping from the redeposited material. The high temperature gradient experienced by outer machined surface results in the existence of thermal and tensile stresses; as a result, the micro-cracks were formed on the machined component.

Figure [3](#page-9-0)(b) depicts the three-dimensional surface topography which describes the height of protrusion and depth of depression on the machined surface at optimized parameters

setting $(A_2B_1C_1D_{-1}E_1)$. In Fig. 3(b), it can be clearly seen that the depth of material removed from the machined surface was approximately 60 µm. In WEDM process, after the removal of material, a crater is formed on the machined due to sparking phenomenon. So, the crater depth is approximately 60 μ m.

5.2 Recast Layer Thickness

Figure [4](#page-10-0)(a) and (b) illustrates the recast layer deposited on the machined surface and elemental composition of the recast layer using EDS at optimized parameters setting $(A_2B_1C_1D_{-1}E_1)$. A recast layer of thickness 19.51 μ m is formed on the machined surface which is inspected by the cross-sectional view of the machined surface through SEM. The elemental composition of the machined surface using EDS reveals the existence of foreign elements such as copper (Cu), zinc (Zn) , carbon (C) , and oxygen (O) from the dielectric and wire electrode. In EDS of machined surface, 4.15% Cu, 0.33% Zn, 2.19% O, and 14.48% C as foreign elements were detected. Apart from the base material (Ni and Ti), the existence of the foreign elements in the recast layer is because of chemical reaction at the elevated temperature. At higher temperature, the foreign elements were diffused to the machined surface.

5.3 XRD Analysis

The XRD peaks for $Ni_{55.95}Ti_{44.05} SMA's machined surface$ at optimized parameters setting $(A_2B_1C_1D_{-1}E_1)$ with the detailed information are illustrated in Fig. [5.](#page-10-0) The identified peaks of XRD divulge the formation of oxides and compounds of Ni, Ti, Zn and Cu, namely NiTi, Ni₄Ti₃, Ti₄O₃, Cu₅Zn₈, Ni(TiO₃), and NiZn on the machined surface. The oxides and compounds of Ni and Ti were formed because of the high reactivity of Ti and Ni atoms. The presence of compounds of Cu and Zn is due to the diffusion of foreign atoms from brass wire and dielectric fluid to the machined surface. The shifting of peaks toward the right side in the XRD pattern indicates the presence of tensile residual stress on machined surface. The results of XRD pattern also support the EDS results of the recast layer.

5.4 Analysis of Micro-hardness

The variation of micro-hardness for the machined surface of Ni_{55.95}Ti_{44.05} SMA at optimized parameters setting $(A_2B_1C_1D_{-1}E_1)$ is displayed in Fig. [6](#page-11-0). It was observed that the micro-hardness in the vicinity of the outer machined surface was increased approximately 1.98 times as compared to the bulk hardness of $Ni_{55.95}Ti_{44.05}$ SMA. This increase in hardness may be due to either oxides formation or quenching effect. In WEDM process, the outer surface is exposed to extremely high temperature and experiences sudden cooling by the pressurized dielectric, which can increase the hardness near the outer surface. The oxide formation (NiTiO₃ and Ti₄O₃) and the diffusion of another alloying element in the recast layer may

Fig. 3 Surface characteristics a SEM micrograph and b 3D surface topography at optimal parameters setting (A2B1C1D-1E1)

Fig. 4 Cross-sectional SEM micrograph a recast layer formed on the machined surface and b EDS of recast layer at optimal parameters setting (A2B1C1D-1E1)

Fig. 5 XRD peaks for machined surface at optimal parameters setting (A2B1C1D-1E1)

also be responsible for hardening effect. Similar hardening effect near the outer machined surface in WEDM process was also observed by Hsieh et al. (Ref [12](#page-11-0)) and Bisaria and Shandilya (Ref [5](#page-11-0)) for NiTi-Cr/Zr ternary SMA and Ni-rich SMA, respectively.

6. Conclusions

In this experimental work, MRR and surface roughness for Ni_{55.95}Ti_{44.05} SMA have been mathematically modeled and analyzed using RSM technique. Surface integrity aspects at the optimized level of parameters were studied. Based on the experimental results and analysis, the following findings can be drawn.

- 1. As per GRA technique, WEDM optimized parameter setting for $Ni_{55.95}Ti_{44.05}$ SMA was $A_2B_1C_1D_{-1}E_1$, i.e., A $(T_{\text{ON}} = 123 \text{ }\mu\text{s})$, B $(T_{\text{OFF}} = 58 \text{ }\mu\text{s})$, C $(\text{SV} = 50 \text{ V})$, D (WT = 3 N), and E (WF = 5 m/min). An increment of 21.73% for MRR and decrement of 9.34% for R_a were achieved compared to initial setting $A_{-1}B_{-1}C_{-1}D_{-1}E_{-1}$, i.e., A $(T_{ON} = 117 \text{ }\mu\text{s})$, B $(T_{OFF} = 54 \text{ }\mu\text{s})$, C $(SV = 40$ V), D (WT = 3 N), and E (WF = 3 m/min). The percentage error between experimental and predicted value was obtained 4.858 and 3.980 for MRR and R_a , respectively, which validates the accuracy of the developed RSM model.
- 2. Micro-structural analysis of machined surface reveals that the machined surface contains micro-voids, micro-cracks, debris, and spherical nodules, whereas the 3D surface

Fig. 6 Micro-hardness variation with the depth from machined surface at optimal parameters setting (A2B1C1D-1E1)

topography shows the depth of crater was approximately $60 \mu m$.

- 3. A recast layer of thickness $19.51 \mu m$ with 21.15% foreign elements (Cu, Zn, O, and C) from brass wire electrode and ionization of dielectric fluid transferred to the machine surface was formed.
- 4. XRD analysis of the machined surface exposes the presence of the compounds of Ni and Ti such as NiTi, $Ni₄Ti₃, Ti₄O₃, Cu₅Zn₈, Ni(TiO₃), and NiZn.$
- 5. Micro-hardness near the machined surface was found to be increased 1.98 times as compared to bulk hardness because of quenching effect and oxide formation.

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