Experimental investigation of MWCNTs mixed EDM of Ti-6AI-4V surface

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Abstract: In the present study, Taguchi's methodology with L₉ orthogonal array applied for the investigation of ED machined Ti-6Al-4V alloy viz. discharge current, pulse-on-time, pulse-off-time and voltage as input process parameters. MWCNTs mixed hydrocarbon oil used as dielectric medium to machine Ti-6Al-4V alloy using graphite electrode with reverse polarity. The output responses assessed as material erosion rate, electrode wear and surface integrity. The significance and contribution of input process parameters on the output responses analysed using analysis of variance. From the experimental results, discharge current evident significant factor for material erosion rate (contribution: 75.76%) and electrode wear rate (contribution: 77.55%). Whereas, pulse-on-time (contribution: 65.75%) followed by pulse-off-time (contribution: 28.18%) exhibits notable surface finish. Furthermore, a regression based model is developed and validated through a confirmative test. The micromechanics of the ED machined surface in terms changed morphology and phase transformation due to thermo process investigated using SEM and XRD analysis respectively.

Keywords: powder mixed electrical discharge machining; PMEDM; material erosion rate; electrode wear rate; EWR; surface integrity; regression model.

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1 Introduction

Ti-6Al-4V alloy possess exceptional metallurgic, materialistic and mechanical characteristics that include strength-over-weight ratio, high thermal stability and superior corrosion resistance compared to other titanium alloys. It is the leading and most pertinent titanium alloy in industries such as aerospace, automotive, chemical and bio-medical (Devgan and Sidhu, 2019). Ti-6Al-4V alloy, known for its lower toughness

and thermal conductivity making it difficult to machine with conventional processes because of higher cutting tool costs incurred (Jabbaripour et al., 2012).

Electrical discharge machining (EDM) is a high-flying non-traditional thermoelectric type machining technique, where material removal is due to high frequency spark generation between two electrical conductive materials, i.e., workpiece and the tool (Sidhu and Bains, 2017; Singh et al., 2019d). However, numerous studies reported that EDM compromises with the surface finish and also possess low material removal rate during machining (Singh et al., 2019b; Bhui et al., 2019). To handle such issues, powder mixed electrical discharge machining (PMEDM) attracted the researchers, by suspending the favoured material to dielectric in powdered form for superior machining stability, augmented discharge frequency, improved material erosion rate, better surface integrity (Bajaj et al., 2015; Kumar et al., 2017; Lamichhane et al., 2019) and for surface modification of the substrate material (Singh et al., 2019c).

Among various researchers reported their work on CNTs mixed EDM, Prabhu and Vinayagam (2010) investigated the influence of single walled CNTs (SWCNTs) in dielectric medium to machine AISI D2 tool steel using EDM. Better surface finish and reduced cracks were observed compared to samples machined in plain dielectric. Dresselhaus et al. (2004) reviewed the electric, thermal, and mechanical characteristics of CNTs. They concluded that CNTs can be used as additive in dielectric medium for the promised properties they possess for efficient ED machining. Sari et al. (2013) demonstrated greater MER and surface finish, reduced EWR, and thinner recast white layer by PMEDM of AISI H13 tool steel and adding MWCNTs in dielectric medium. Izman et al. (2012) investigated the effect of MWCNTs mixed dielectric medium on Ti-6Al-4V alloy with copper tool. Around 7% and 9% improvement in MER and surface roughness (SR) was resulted, along with reduced white recast layer compared to specimen machined without MWCNTs mixed dielectric. Kolli and Kumar (2016) optimised the concentration of graphite powder (14 g/l) and surfactant (6 g/l) in dielectric medium for the ED machining of Ti-6Al-4V alloy with copper electrode. From the results, better MER and smoother surface, reduced electrode wear, along with thinner recast white layer was detected.

Shabgard and Khosrozadeh (2017) through his research paper investigated the effect of MWCNTs (2 g/l) mixed PMEDM of Ti-6Al-4V alloy with copper tool. They observed enhanced metal removal, reduced electrode wear, better surface finish with reduced micro-cracks, and improved machining stability, compared to pure dielectric machining. Bhui et al. (2018) through their research explored the optimal EDM parameters for machining Ti-6Al-4V alloy with graphite electrode. Findings revealed better material removal and porous surface. SEM and XRD showed the bioactive surface which was further validated with appetite growth revealed by EDS analysis. Bains et al. (2019) used nano-hydroxyapatite (HAp) mixed in dielectric medium to achieve the desired surface modification of Ti-6Al-4V alloy surface applying EDM process. SEM and XRD revealed the deposits of calcium and phosphate on the treated surface. Improved corrosion and wear rates of the modified surface were resulted. The surface modification of β -type Ti alloy (Ti-Nb-Ta-Zr) was carried out by Devgan and Sidhu (2020) using MWCNTs and HAp nano powders separately mixed in dielectric medium using electric discharge machining. MWCNT treated surface resulted in a more favourable biocompatible surface with improved cell adhesion and proliferation of blood and tissue cells. The cytotoxicity of the treated surface improved by around 95.3%.

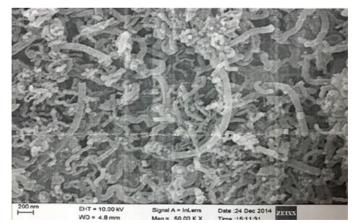
From the literature, multi-walled carbon nanotubes (MWCNTs) noteworthy improves the machining rate and surface properties of substrate material. The current proposed work is devoted to investigate the performance of MWCNTs powder mixed EDM on grade 5 titanium alloy with graphite as tool electrode. The reverse polarity is opted for the research study to scrutinise the surface modification of the substrate, providing better material transfer rate during machining. The output responses measured in material erosion rate, electrode wear rate (EWR), surface finish of the machined surface and analysed statistically via. ANOVA. Additionally, regression equations developed and validated by comparing the predicted and experimental values.

2 Experimental work

2.1 Materials

The workpiece material, Ti-6Al-4V alloy in the form of plate ($160 \text{ mm} \times 80 \text{ mm} \times 5 \text{ mm}$) having chemical composition of Ti: 89.546%; Al: 6.1%; V: 4.2%; Fe: 0.09%; C: 0.03%; O: 0.03%; N: 0.003; H: 0.001% procured from Baoji Fuyuantong Industry & Trade Co. Ltd., China. Electrolytic graphite tool with diameter 10 mm; density 2.26 g/cm³; melting point 3,650°C and thermal conductivity of 24 W/m.K from Mersren, Germany was chosen as electrode in the present study. The emery papers of grit size 1,000, 1,200 and 2,000 used to smoothen the face of electrode before each experimental run. MWCNTs mixed at 7 g/l in the hydrocarbon base dielectric medium to amplify the machining process and surface integrity. Table 1 listed the MWCNTs properties and description, whereas SEM of nanotubes in Figure 1 (source: technical data sheet, provided with powder) purchased from United Nanotech, Bangalore, India.

Figure 1 SEM image of as-received MWCNTs



2.2 Methodology

Taguchi's technique of orthogonal array utilised to reduce the number of experimental runs that needed to frame the model of experimentation perform compared to the full factorial design of experiments. In the present study, L_9 orthogonal array employed with the help of statistical Minitab-17 software considering discharge current (Dc), pulse-on-time (P-on), pulse-off-time (P-off) and voltage (V) as input machining factors, each factor having three levels, i.e., small, medium and large, as shown in Table 2.

Property	Description	
Production method	Chemical vapour deposition	
Available form	Black powder	
Diameter	Outer diameter: 10-30 nm	
Length	10 micron	
Nanotubes purity	>95%	
Metal particles	<4%	
Amorphous carbon	<1%	
Specific surface area	330 m ² /g	
Bulk density	0.04–0.06 g/cm ³	

Table 1Physical properties of MWCNTs

Source: Technical data sheet, provided with powder

 Table 2
 Input machining parameters

Factors	Units	Level 1	Level 2	Level 3
Discharge current (Dc)	Ampere	1	2	4
Pulse-on time (P-on)	µ-seconds	30	45	60
Pulse-off time (P-off)	µ-seconds	60	90	120
Voltage (V)	Volt	30	40	50

 Table 3
 Experimental design based on L9 orthogonal array

Exp. no.	Dielectric	Dc	P-on	P-off	V
1	EDM oil + MWCNTs	1	30	60	30
2	EDM oil + MWCNTs	1	45	90	40
3	EDM oil + MWCNTs	1	60	120	50
4	EDM oil + MWCNTs	2	30	90	50
5	EDM oil + MWCNTs	2	45	120	30
6	EDM oil + MWCNTs	2	60	60	40
7	EDM oil + MWCNTs	4	30	120	40
8	EDM oil + MWCNTs	4	45	60	50
9	EDM oil + MWCNTs	4	60	90	30

Each combination of listed experiments in Table 3 performed twice and an average of both was used to minimise the error and noises for precise output responses. The depth of cut was kept constant to 1 mm for all experimental trials.

2.3 Experimentation

A total of 18 experimental run (9 × 2 runs) was performed on die-sinker type EDM machine, model OSCARMAX S645 CMAX, make Taiwan with hydrocarbon oil (commercial EDM oil with specific gravity = 0.763, freezing point = 94°C) mixed MWCNTs as dielectric medium. Instead of using EDM tank, an indigenously developed tank was used of capacity 15 litres and dimensions $12'' \times 9'' \times 9''$ as shown in Figure 2. A stirrer for uniformly mixing and to avoid the settling down of powder in dielectric was placed on the top of container. Additionally, a pump was installed in this tank setup for the appropriate flow and adequate flushing of debris from the machined surface. Furthermore, surface morphology and compositional analysis of the machined samples were examined using SEM (JSM-6610 LV Joel, Japan) and XRD (X'pert Pro, Netherland) respectively.

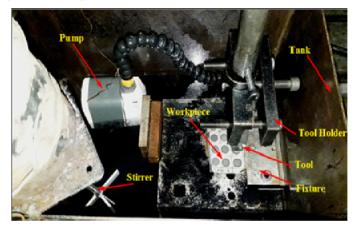


Figure 2 Experimental setup for MWCNTs mixed PMEDM (see online version for colours)

3 Results and discussion

Based upon the experimentation performed, the measurements were obtained in terms of material removal from the workpiece and tool electrode, and roughness of the machined surface for the further calculation of the output variables. Table 4 shows the results of output response values for both the workpiece plates along with their signal-to-noise ratios (S/N ratios).

MER and EWR were calculated using the equation (1):

$$MER / EWR = \frac{Initial weight - Final weight}{Machining time} \times 1,000 \text{ mg} / \text{min.}$$
(1)

3.1 Effects of machining parameters on metal erosion rate (MER)

Figure 3 showed the average variation of S/N ratios at different levels of input parameters according to 'larger-is-better' criteria for MER, where the larger value of S/N ratio

represents the maximum material erosion value during the machining process. Maximum erosion rate of 23.895 mg/min. was observed at discharge current 4 A, pulse-on 45 μ s, pulse-off 60 μ s and voltage 50 V. It is evident from the previous studies that the applied current intensify the material erosion from the workpiece material during the EDM process (Kliuev et al., 2019; Singh et al., 2019a). Consequently, drastic amplification in erosion rate was observed for higher peak current (4 A) compared to 1 A with an increment of 403.5% in the present study. Similar results can be validated from S/N ratios plot that erosion rate was directly proportional to the applied current intensity. Figure 3 also portrays the continuous increment of MER with respect to the value of pulse-on-time from 30 μ s to 60 μ s. However, the random deviation of S/N ratios with respect to change in pulse-off-time and voltage levels could not reflect any significant affect on the MER. The average of each level in terms of S/N ratios and the rank of process parameters is tabulated in Table 5.

Exp.	MER (n	ng/min.)	S/N ratio	EWR (n	ng/min.)	S/N ratio	SR ((µm)	S/N ratio
no.	<i>R1</i>	R2	(MER)	<i>R1</i>	R2	(EWR)	<i>R1</i>	R2	(SR)
1	2.86	3.00	9.3299	0.84	0.72	2.1325	0.318	0.357	9.4201
2	3.93	3.86	11.8091	0.67	0.98	1.5203	0.263	0.246	11.8814
3	5.14	4.63	13.7418	1.02	0.91	0.2827	0.203	0.170	14.5526
4	4.79	5.04	13.8220	0.97	1.09	-0.2635	0.343	0.390	8.7007
5	5.78	5.71	15.1853	1.29	1.13	-1.6747	0.396	0.346	8.5928
6	16.11	16.27	24.1846	2.52	2.84	-8.5781	0.066	0.079	22.7585
7	11.98	11.63	21.4385	2.29	2.06	-6.7613	0.487	0.466	6.4366
8	24.27	24.52	27.5572	3.64	3.58	-11.1504	0.178	0.169	15.2111
9	23.65	22.89	27.3325	3.70	4.13	-11.8677	0.189	0.174	14.8151

 Table 4
 Response table of EDMed Ti-6Al-4V in MWCNTs mixed dielectric

Notes: R1 and R2 are repetitions of experimentations on two separate plates.

Level	Current	Pulse-on	Pulse-off	Voltage
1	11.63	14.86	20.36	17.28
2	17.73	18.18	17.65	19.14
3	25.44	21.75	16.79	18.37
Delta	13.82	6.89	3.57	1.86
Rank	1	2	3	4

Table 5Response table for S/N ratios for MER

3.2 Effects of machining parameters on EWR

Figure 4 and Table 6 illustrates the main effects plot and response table for S/N ratios of EWR, respectively using 'smaller-is-better' criterion. From the graph (Figure 4), it is observed that as the discharge current and pulse-on duration increased, the electrode wear also increases. According to the chosen principle of 'smaller-is-better', the minimum value of EWR reflects corresponds to the maximum value of S/N ratio and vice-versa. The machining parameters at the settings of their lower levels, i.e., discharge current 1 A,

pulse-on 30 μ s, pulse-off 60 μ s, voltage 30 V depicts minimum electrode wear (0.780 mg/min.).

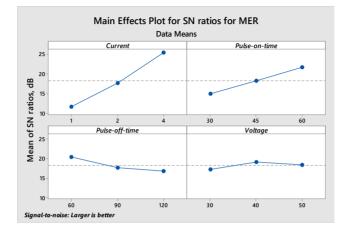


Figure 3 S/N ratios plot for effects of machining parameters on MER (see online version for colours)

Figure 4 S/N ratios plot for effects of machining parameters on EWR (see online version for colours)

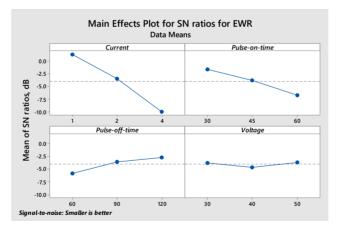


Table 6Response table for S/N ratios for EWR

Level	Current	Pulse-on	Pulse-off	Voltage
1	1.312	-1.631	-5.865	-3.803
2	-3.505	-3.768	-3.537	-4.606
3	-9.926	-6.721	-2.718	-3.710
Delta	11.238	5.090	3.148	0.896
Rank	1	2	3	4

3.3 Effects of machining parameters on SR

Figure 5 exhibited the S/N ratio graph for SR, prepared as per the 'smaller-is-better' criterion, where maximum S/N ratio indicate the minimum roughness value. From the graph, it is observed that the pulse-on-time is most dominant factor followed by pulse-off-time for the roughness of ED machined surface. Similar findings revealed by the delta values and respective ranks of machining parameters for S/N ratios of SR as showed in Table 7. The random variation of SR with respect to discharge current results due to the participation of carbon particles in the working zone. The superior surface finish of 0.0725 μ m observed at parametric settings of current 2 A, pulse-on 60 μ s, pulse-off 60 μ s, voltage 40 V.

Figure 5 S/N ratios plot for effects of machining parameters on SR (see online version for colours)

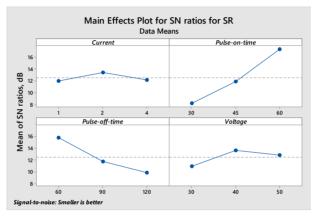


Table 7Response table for S/N ratios for SR

Level	Current	Pulse-on	Pulse-off	Voltage
1	11.951	8.186	15.797	10.943
2	13.351	11.895	11.799	13.692
3	12.154	17.375	9.861	12.821
Delta	1.399	9.190	5.936	2.750
Rank	4	1	2	3

3.4 Analysis of variance for MER, EWR and SR

The output responses were further analysed statistically using Mintab-17 software to determine the significant parameters and presented in Table 8. For ANOVA, the insignificant factors are pooled, and the dominance of significant parameters analysed using their p-value (< 0.05) and relative contributions of the selected parameters. The most influencing parameter for material erosion rate is discharge current with p-value 0.018 and contribution of 75.76%. Also, the discharge current (p-value: 0.008; contribution: 77.55%) along with pulse-on-time (p-value: 0.036; contribution: 15.94%) considerably involve in the EWR. However, the applied current examined as insignificant for the SR and only pulse-on-time turns to be significant with p-value 0.026 and contribution of 65.75%.

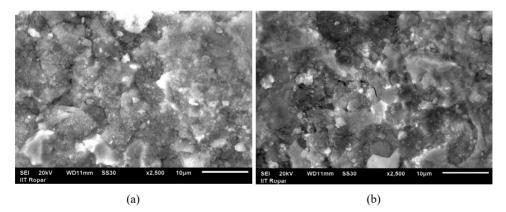
Table 8	Analysis	of variance	for output responses
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Doute Dr MER EWR SR MER EWR SR MER EWR EU038 Dise Dis Dise Dise D	⁷ ariance		p-value		%	% contribution	ио
scurrent 2 287.60 190.73 # 143.80 95.36 # 2 71.22 39.19 128.24 35.61 19.59 64.12 2 20.79 16.00 54.97 10.39 7.99 27.48 2 # # 11.84 # 5.92 5.24 1.45 3.43 2.62 0.72 1.71 3.8487 7.04 108.40		MER	EWR	SR	MER	MER EWR	SR
2 71.22 39.19 128.24 35.61 19.59 64.12 2 20.79 16.00 54.97 10.39 7.99 27.48 2 # # 11.84 # # 5.92 5.24 1.45 3.43 2.62 0.72 1.71 3.8487 2.40 108.40		0.018*	0.008*	#	75.76	77.55	
2 20.79 16.00 54.97 10.39 7.99 27.48 2 # # 11.84 # # 5.92 5.24 1.45 3.43 2.62 0.72 1.71 384.87 247 30 108.40		0.069	0.036^{*}	0.026^{*}	18.76	15.94	65.75
2 # # 11.84 # # 5.92 5.24 1.45 3.43 2.62 0.72 1.71 384 87 247 30 108 40		0.202	0.083	0.059	5.48	5.48 6.51	28.18
5.24 1.45 3.43 2.62 0.72 384 87 247 30 108 40		#	#	0.225	#	#	6.07
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3.5 SEM analysis of the machined surface

The machined surface was then examined using scanning electron microscopy to investigate its changed surface morphology. During the ED machining process, the elevated temperature in the working area vaporised the material and results in micro-cracks, craters and splashes of molten droplets on the surface (Bhaumik and Maity, 2018; Rajeswari and Shunmugam, 2019; Mandal and Mondal, 2019). Figure 6 shows the SEM micrograph of Ti-6Al-4V alloy machined surface exhibiting minimum material erosion (trial 1) and maximum value of material erosion rate (trial 8). Both the SEM images reveal crater free surface due to the presence of powder particles deposited on the machined surface during the ED machining in the powder mixed dielectric. However, the surface machined (trial 1) at low value of discharge current and pulse-on-time depicts more splashes of molten metal because of the smaller amount discharge energy produced within the working area. Comparatively, trial 8 portrays a uniform porous surface with some micro-cracks over it. The generation of higher spark energy in trial 8 causes the proper melting of the working zone, which helps in the deposition of powder particles on it by striking through flushing nozzle and sticking within. The surface thus produced with porous texture could prove the proper cell growth and bone-implant adhesion when used as biomaterial (Mahajan and Sidhu, 2019; Manoj et al., 2019).

Figure 6 SEM showing, (a) machined surface for minimum MER (trial 1) (b) surface with maximum MER (trial 8) (see online version for colours)



3.6 XRD analysis of the machined surface

The compositional alternations of the ED machined surface was disclosed using X-ray diffraction. Figure 7 shows the XRD spectra of the sample machined at higher value of discharge current (trial 8), as the maximum possibility of the phase transformation and intermetallic compound formation would have occurred under these conditions.

From the XRD peaks, the surface machined with the addition of MWCNTs in the dielectric is found crowded with inter-metallic compounds, oxides and carbides as shown in figure.

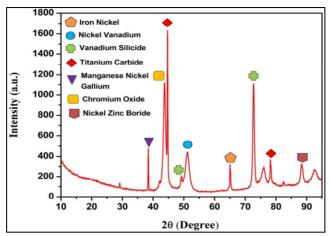


Figure 7 XRD spectra for trial 8 (see online version for colours)

3.7 Regression analysis model

Regression model analysis was accustomed for the correlation of selected machining parameters and the output response variables. The model was generated using the Minitab-17 statistical software and the equations for material erosion rate [equation (2)], EWR [equation (3)] and SR [equation (4)] are as follows:

$$MER = -4.67 + 5.266 \text{ Dc} + 0.2744 \text{ P-on} - 0.11433 \text{ P-off} + 0.0292 \text{ V}$$
(2)

$$EWR = -0.1702 + 0.7927 Dc + 0.03976 P-on - 0.015100 P-off - 0.00498 V$$
(3)

$$SR = 0.502 + 0.0075 Dc - 0.00822 P - on + 0.00249 P - off - 0.00271 V$$
(4)

where 'Dc' is the discharge current in (Ampere), P-on is pulse-on-time in (μ s), P-off is pulse-off-time in (μ s) and gap voltage (V).

Exp.	M	ER	$C(\theta_{\ell})$	EV	VR	$C(\theta/)$	S	'R	$C(\theta/)$
no.	A	В	C (%)	A	В	- C (%)	A	В	C (%)
1	2.930	2.844	97.07	0.780	0.760	97.42	0.3375	0.3310	98.07
2	3.895	3.822	98.13	0.825	0.854	96.54	0.2545	0.2553	99.68
3	4.885	4.800	98.26	0.967	0.947	97.94	0.1865	0.1796	96.30
4	4.915	5.264	92.89	1.029	1.000	97.18	0.3665	0.3590	97.95
5	5.745	5.366	93.40	1.210	1.243	97.27	0.3710	0.3646	98.27
6	16.190	16.634	97.25	2.680	2.696	99.41	0.0725	0.0648	89.37
7	11.805	12.074	97.71	2.175	2.182	99.66	0.4765	0.4758	99.85
8	23.895	23.342	97.68	3.610	3.635	99.31	0.1735	0.1760	98.55
9	23.270	23.444	99.25	3.915	3.878	99.04	0.1815	0.1816	99.94

Table 9Result for confirmation test

Notes: A = mean experimental values of R1 and R2; B = predicted value; C = agreement with prediction (%).

3.7.1 Confirmation test

To validate the predicted values compared to the actual experimental results, confirmatory test performed. It is observed that the regression results closely predicted the response values to the actual experimentally measured MER, EWR and SR with minimum error. On the grounds of confirmatory test comparison (Table 9), developed model has higher potency to inspect the output responses of ED machined Ti-6Al-4V in MWCNTs mixed dielectric medium.

4 Conclusions

Following conclusions were drawn based on the present investigation of powder mixed ED machined Ti-6Al-4V alloy with graphite tool in MWCNTs added dielectric medium:

- The most significant factors in terms of achieving maximum MER was discharge current (contribution: 75.76%), followed by pulse-on-time (contribution: 18.76%). Maximum erosion rate of 23.895 mg/min. was observed at discharge current 4 A, pulse-on-time 45 µs, pulse-off-time 60 µs, voltage 50 V.
- The dominating parameter for attaining minimum EWR was discharge current (contribution: 77.55%) followed by pulse-on-time (contribution: 15.94%) and pulse-off-time. Minimum EWR (0.780 mg/min.) was observed at discharge current 1 A, pulse-on-time 30 μs, pulse-off-time 60 μs, voltage 30 V.
- Superior surface finish (0.0725 μm) was observed for discharge current 2 A, pulse-on-time 60 μs, pulse-off-time 60 μs, voltage 40 V.
- SEM analysis of the EDMed surface exhibited splashes of molten metal, micro-cracks and formation of homogeneous micro-pores that were evenly distributed on the surface.
- XRD analysis revealed that the surface machined with greater spark energy illustrates inter-metallic compounds, oxides and carbides.

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