Material characterization and unconventional machining on synthesized Niobium metal matrix

C Sivakandhan 1, Ganesh Babu Loganathan 2, G Murali 3, P Suresh Prabhu 4, S Marichamy 5, G Sai Krishnan 6 and Raghuram Pradhan 7

1 Department of Mechanical Engineering, Sri Indu Institute of Engineering and Technology, Hyderabad, Telangana 501 510, India
2 Department of Mechatronics Engineering, TISHK International University, Erbil, KRG, Iraq
3 Department of Mechanical Engineering, Konera Lakshmaiah Education Foundation, Guntur, Andhra Pradesh 522 502, India
4 Department of Mechanical Engineering, Karpagam University, Coimbatore, Tamil Nadu 641021, India
5 Department of Mechanical Engineering, Sri Indu College of Engineering and Technology, Hyderabad, Telangana 501 510, India
6 Department of Mechanical Engineering, Rajalakshmi Institute of Technology, Chennai, Tamil Nadu, India
7 Department of Mechanical Engineering, Pace Institute of Technology and Sciences, Ongole, Andhra Pradesh, India

E-mail: g.saikrishnan@gmail.com

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Abstract

The Purpose of this work is to develop a niobium based metal matrix alloy through sintering based powder metallurgy technique. The 2, 4 and 6 weight percentage of Titanium Carbide (TiC) is added to the alloy. Various material properties such as hardness, tensile strength, impact strength and density are measured after the addition of TiC particle. The characterization, microstructure and particle size of the developed metal matrix are learnt through Scanning Electron Microscope (SEM) and Energy Dispersive Analysis of x-rays (EDAX). Finally, the synthesized niobium metal matrix is machined by unconventional machining processes namely Ultrasonic Machining (USM) and Laser Beam Machining (LBM) processes. The various input and output parameters have been considered for this experimental work. The most influential parameters on Material Removal Rate (MRR) and Surface Roughness (SR) are found by Analysis of variance. The systematic evaluation of optimal parameters of MRR and SR are carried out by using taguchi approach.

Nomenclature

SEM Scanning Electron Microscope
EDAX Energy Dispersive Analysis of x-rays
TiC Titanium Carbide
ANOVA Analysis of Variance
USM Ultrasonic machining process
MRR Material Removal Rate
SR Surface Roughness
BHN Brinell hardness number
LBM Laser Beam Machining

1. Introduction

In recent days, the gradual increase of research work associated to metal matrix is apparent because of the great demand of industrial needs. Due to high strength to weight ratio, metal matrixes are preferred in industrial applications. Normally, metal matrix composites are fabricated only by some alloying elements that are added to the base metal. The material properties can be enhanced when adding more alloying elements to the base metal.
Due to its excellent material properties, the niobium metal matrix is used in various areas such as nuclear reactor, aerospace, marine, missiles, jets and production of cutting tools. The homogeneous mixture and excellent material properties were easily achieved in sintering based powder metallurgy technique [1, 2]. The strengthening mechanism and grain refinement were studied on titanium metal matrix which was fabricated through powder metallurgy technique [3]. The material properties such as strength, hardness and density were increased due to the addition of titanium carbide. The various alloying elements such as aluminium, titanium, chromium and molybdenum were used to prepare the nickel metal matrix. Due to its excellent properties, nickel alloys were used in various fields namely nuclear power plant, oil refinery plant, marine and aerospace. Nickel based alloys have good thermal conductivity, corrosion resistance and wear resistance. The powder metallurgy technique was used to fabricate many components with different shape and size [4–8]. The material properties and structure was most affected by reinforcement particles [9]. Alumina and graphite reinforcement particles were used in nickel metal matrix [10, 11]. The frictional wear test was carried out on nickel metal matrix and it was concluded that reinforcement particles were used to reduce the friction coefficient [12, 13]. Tribological experimental investigation was carried out on nickel metal matrix composite with reinforcement of graphite and TiC particles [14]. In ultra sonic machining process, the machining tool was vibrated due to the conversion of ultra sonic sound to mechanical vibrations. The frequency of the electrical power was the most important factor to produce the vibration of the tool. It is evident that very hard materials and metal composites are easily machined by the ultrasonic machining process. In rotary ultrasonic machining process, ceramic metal matrix composites were machined and the responses namely metal removal rate and surface roughness were found [15]. The recovery of the niobium material was extracted from liquid to liquid was studied [16]. Niobium powder was prepared through electronically mediated reaction method [17]. The effect of shape-memory concept was studied in niobium material with different stress conditions [18]. In laser beam cutting process, good quality surface was obtained at lower cutting speed [19]. In laser beam machining process, the molten metal was formed and vaporized from work piece. It can be easily removed by high pressure gas flow jet [20]. The laser beam was used to melt the material up to the vaporization point and it releases from the hole in the form of material vapor [21]. The quality of laser cutting depends on the gas pressure and laser power [22]. Better surface quality can be achieved by pulsed mode type of laser beam [23].

The laser drilling operation was carried out on nimonic alloy and their characteristic of spatter formation was studied [24]. The circularity of the holes mainly depends on the nozzle diameter and gas pressure [25]. The holes geometry was studied in Nd:YAG laser drilling of mild steel sheets [26]. Thermal oriented problems were analyzed in laser micro drilling of thin metal sheets. The molten layer thickness was studied in laser drilling of metals [27]. In W-Ni-Cu composite, nickel plays an important role and it was used to improve material properties like as ductility, ferromagnetism and corrosion resistance [28]. The various input parameters namely power, pulse frequency, pulse width and air pressure were considered during laser drilling of zirconium based ceramics [29]. The response parameters were decided through developed mathematical model [30]. The determined optimal parameters were used to eliminate the uncontrolled parameters [31]. The statistical design of experiment was used to correlate the control and response parameters. The mathematical model was also used to predict the responses [32]. High amount of cutting force was required for conventional machining process. Sometimes thermal damages were also produced. In conventional machining process, larger tool wear and low material removal rate were achieved due to the hardess of the material and the nature of reinforcement particles [33–35]. The machinability, surface quality and tool wear of the metal matrix composites were investigated through ultrasonic and laser machining processes [36]. The micro-structural, mechanical, electrochemical behaviors and thermal properties were analyzed in aluminium metal matrix. The various manufacturing processes and industrial applications were also discussed [37]. The material properties of Al-Si alloy was investigated and its hardness was increased when more addition of silicon carbide particles [38]. Temperature, size of particle and sintering time was controlled and these effects were studied in fabrication of Aluminium Metal Matrix Composites [39]. Analysis of variance was applied in wear experimental investigation on copper with Multi-Walled Carbon Nanotubes (MWCNT) and concluded that the content of MWCNT was the significant factor (76.48%) on wear loss [40]. The material characterization and machinability was studied by varying the boron nitride (BN) in copper which was fabricated through friction stir casting process [41]. By performing the friction stir processing technique a very good percentage of increase in the wear is observed due to its hardness property and self lubricity. Magnesium metal matrix with reduced graphene oxide was analysed through Taguchi coupled grey relation analysis to analyse the machining parameters. A novel aluminium alloy were fabricated and analysed for various healing purpose. Various parameters such as Pulse On time (PON), Pulse Off time (POFF), wire feed rate (WFR) along with the material elemental composition parameters Sn wt% and SiC wt% using Taguchi coupled Grey Relational Analysis were carried out [42–44].

The present research work clearly describes about the various topics such as material synthesis using powder metallurgy technique, evaluation of material properties and unconventional machining processes. The material characterization and microstructure was also studied. The most influential machining parameters were found
by analysis of variance. The optimal parameters of the responses such as MRR and SR are found by taguchi approach.

2. Materials and methods

2.1. Synthesis of niobium metal matrix
The mixing and uniform sizing of the powders were achieved by miller unit. It consists of cylindrical chamber, shaft and impellers. The chamber was filled by various sizes of the metal powders and subsequently the shaft was rotated by an electric motor. All metal powders were agitated by impeller. The speed of the miller is 400 rpm and the powder particles are cold welded. The homogenous mixture and uniform size of the particles were obtained.

After that, the uniform sizes of the particles were processed in to a compact unit. Dies were cleaned to avoid the contamination of the powders. To attain easy removal of specimen from the die unit, acetone paste and zinc oxide was applied. All metal powders were placed in to the die unit and compact pressure of 270 Mpa was applied. The uniform pressure was applied to the die unit and the specimen was removed after 15 min.

2.2. Sintering process
An electric furnace was used in sintering process. The specimen was kept inside the furnace after the compact process. The impurities and moisture contents were removed before starting the process. The temperature of the furnace was gradually increased at the rate of 25 °C per minute. Finally it was reached around 1800 °C. After 30 min, the temperature of the furnace was stopped and allowed to cool in room temperature. The specimen was subjected to material test and characterization after sintering process. The niobium metal matrix was fabricated with 2, 4 and 6 weight percentage of TiC. The samples of niobium metal matrix are shown in figure 1.

3. Characterization of materials
The figure 2(a) shows the SEM image and it was used to study the morphology of the niobium metal matrix. All particles were dispersed on the material which is shown in white color region. The enlargement view of the TiC is also shown in SEM image. The nature of particle reinforcement, size of particles and addition of more alloying elements were used to achieve the favorable microstructure and material properties. The micro structural properties and micro hardness were analyzed in metal matrix. The grain boundary strength was increased due to an addition of chromium, titanium carbide and niobium particles. The EDAX image was shown in figure 2(b). It confirms the occurrence of alloying elements and its composition.

4. Results and discussion

4.1. Properties of niobium metal matrix
The material properties such as hardness, tensile strength, impact strength and density were measured. The mechanical properties mainly depend on the percentage of reinforcement particles which were added in to the composite. Brinell hardness tester, universal testing machine, izod testing machine and Archimedes...
principle was used to measure the hardness, tensile strength impact strength and density of the metal matrix respectively. The various alloying elements such as nickel, silicon, aluminium, niobium, chromium are added. The reinforcement material plays an important role in material properties. Along with three specimens, third have better material properties (6 weight percentage of TiC). The material properties such as hardness (810BHN), tensile strength (780 Mpa), impact strength (19 J) and density (9.8 g cc\(^{-1}\)) were measured for the third specimen. The Density of the developed composites was measured with the help of the Archimedes principle. Based on the results it can be concluded that the 6 Weight percentage of TiC possessed the highest value of 9.8 g cc\(^{-1}\). Increasing the TiC content with Niobium increased the density of the developed composite. This is mainly due to the percentage of niobium and titanium percentages present. This is attributed due to the dense percentage of titanium carbide and ultra low density of niobium, which is in tandem with the literature [21]. Based on the hardness values obtained Increasing the volume fraction of the TiC increased the hardness to 810BHN. Based on the literature review present Increase in the hardness value is due to the uniform distribution of TiC with Niobium. The 6 weight percentage of TiC had the highest hardness value. Similary the Same trend was observed for tensile strength and impact strength. Since on comparing all the three composites 6 weight percentage of titanium carbide had the better result third specimen is considered for these experimental investigations[22–24].

4.2. Ultrasonic machining of metal matrix
An ultrasonic machining is the most suitable method for machining of advanced materials and hard materials [51]. USM is the stress free process which depicts good surface structure [52]. The ultra sonic machining of niobium metal matrix (6 wt% of TiC) was carried out by SONIC—500 model. During machining process, frequency of vibration (25 KHz), static load (1.5 Kg), amplitude of vibration (20 \(\mu\)m), thickness of work piece (8 mm), slurry temperature (30 °C) and slurry (Al\(_2\)O\(_3\) + SiC) were considered. In ultrasonic machining, input parameters are power rating (100–300 W), slurry concentration (20%–40%) and grit size (300–500). The output parameters are Material Removal Rate (MRR) and Surface Roughness (SR). Ultrasonic machining experimental observations were shown in table 1. In ultra sonic machining process, MRR was increased by ultra sonic vibrations.

4.3. Analysis of variance (ANOVA) for MRR and SR
ANOVA is carried out for determining percentage contribution of each parameter. From table 2, it is very clear that power rating is the most contribution factor on material removal rate due to the production of ultrasonic vibrations. The percentage of slurry concentration provides the most contribution factor on surface roughness due to the action of abrasive slurry.

5. Contour plot analysis for MRR and SR
Figures 3(a)–(c) shows that contour plot analysis for MRR. The higher material removal rate was shown in dark region. Other region shows the moderate and lower material removal rate. Figures 3(d)–(f) shows the contour
plot analysis for Sr. The good surface roughness was shown in white region. Other region shows that moderate and poor surface roughness [53, 54].

5.1. Optimal parameters
The objective of the material removal rate is maximum and surface roughness is minimum. Based on these conditions, the optimal parameters were found by taguchi approach. Figure 4(a) shows the mean of Signal to Noise (S/N) ratios for MRR and Sr. After finding out the mean of signal to noise ratio, main effect plots were drawn for MRR and SR which was shown in figures 4(b), (c). Larger the better criteria was considered for MRR. From figure 4(b), the maximum material removal rate was attained at power rating (300 W), percentage of slurry concentration 40. A summary of the experimental observations is shown in Table 1. The analysis of variance for MRR is shown in Table 2. The analysis of variance for SR is shown in Figure 3. Table 1. Ultra sonic machining experimental observations.

| S. No | Power rating (W) | Slurry concentration (%) | Grit size | MRR (mg min⁻¹) | SR (microns) |
|-------|-----------------|--------------------------|-----------|----------------|--------------|
| 1     | 100             | 20                       | 300       | 10.34          | 0.64         |
| 2     | 100             | 30                       | 400       | 10.56          | 0.54         |
| 3     | 100             | 40                       | 500       | 11.45          | 0.87         |
| 4     | 200             | 20                       | 400       | 11.11          | 0.76         |
| 5     | 200             | 30                       | 500       | 11.78          | 1.23         |
| 6     | 200             | 40                       | 300       | 09.23          | 5.57         |
| 7     | 300             | 20                       | 500       | 11.01          | 0.87         |
| 8     | 300             | 30                       | 300       | 12.45          | 1.43         |
| 9     | 300             | 40                       | 400       | 13.12          | 1.78         |

Table 2. Analysis of variance for MRR.

| Source                  | DF | Adj. SS | Adj. MS | F-Value | P-Value % | Adj. SS | Adj. MS | F-Value | P-Value % | Adj. SS | Adj. MS | F-Value | P-Value % |
|-------------------------|----|---------|---------|---------|-----------|---------|---------|---------|-----------|---------|---------|---------|-----------|
| Power rating (W)        | 2  | 4.204   | 2.10    | 1.01    | 0.49      | 39.24   | 5.177   | 2.588   | 1.74      | 0.365   | 26.25   |
| Slurry concentration (%)| 2  | 0.911   | 0.45    | 0.22    | 0.82      | 08.50   | 6.830   | 3.415   | 2.29      | 0.304   | 34.63   |
| Grit size               | 2  | 1.433   | 0.71    | 0.34    | 0.74      | 13.38   | 4.735   | 2.367   | 1.59      | 0.386   | 24.01   |
| Error                   | 2  | 4.165   | 2.08    | —       | —         | 38.88   | 2.978   | 1.489   | —         | —       | 15.11   |
| Total                   | 8  | 10.71   | —       | —       | —         | 100     | 19.72   | —       | —         | —       | 100     |

Figure 3. (a)–(f). Contour plot analysis for MRR and Sr.
concentration (30%) and grit size (400). Smaller the better criterion was considered for Sr. From Figure 4(c), the minimum surface roughness was attained at power rating (200 W), percentage of slurry concentration (40%), and grit size (300).

6. Laser beam machining of metal matrix

Laser beam machining (LBM) is one of the thermal energy-based effective unconventional machining processes which are broadly used in manufacturing industries. The niobium metal matrix was drilled by LBM process. In laser drilling process, laser power, cutting speed, and gas pressure were considered as the main control parameters. The material removal rate (MRR) and surface roughness (SR) are considered as the response parameters. The experiment has been validated using Analysis of Variance (ANOVA). The use of laser beam machining process has been increased in all kinds of industries due to its high accuracy and precision [54]. The high amount of thermal energy was produced through laser beam. In this process, tool and work piece were not in direct contact with each other [49]. The high amount of energy is used to heat and melt the work material. There is no need of cutting tool, work holding devices, and mechanical forces in LBM [50]. By varying the control parameters namely laser power, cutting speed, and gas pressure, the output parameters such as MRR and SR are recorded and shown in the table 3.

6.1. Analysis of variance for MRR

From Table 4, it has been observed that cutting speed is the significant factor followed by gas pressure and laser power. The p-values less than 0.0500 indicate that the model terms are significant. In this investigation, cutting speed and gas pressure are found to be significant model terms. The interactions of B and C are found to be significant. F-value of 0.19 implies that the Lack of Fit is not significant relative to the pure error. There is an 89.36% chance that a large F-value could occur due to noise. Here A, B, and C are the cutting speed, laser power, and gas pressure, respectively.

The predicted $R^2$ (0.634) is in reasonable agreement with the Adjusted $R^2$ (0.808) i.e., the difference is less than 0.2. Adequate precision value (9.183) indicates an adequate signal. The value of $R^2$ (adjusted) with 80.8% accounts for the number of predictors in the model which may get smaller with the accumulation of terms.
6.2. Analysis of variance for SR

From Table 4, it has been observed that laser power is the most significant factor for SR with p-values less than 0.05, indicating that the model terms are significant. In this investigation, interaction effect of cutting speed and gas pressure is the significant model term. The predicted $R^2 (0.8637)$ is in reasonable agreement with the adjusted $R^2 (0.9532)$, i.e., the difference is less than 0.2. This model can be used to navigate the design space. The value of $R^2 (adjusted)$ with 95.32% accounts for the number of predictors in the model, which may get smaller with the accumulation of terms.

7. Conclusion

After successful completion of experimental investigations of niobium metal matrix, the following findings were concluded.

- The niobium metal matrix with 2, 4 and 6 weight percentage of TiC was successfully fabricated through powder metallurgy route. Among three specimens, third one has better material properties.

- The material properties such as hardness (810 BHN), tensile strength (780 Mpa), impact strength (19 J) and density (9.8 g cc$^{-1}$) were measured. The hardness value was increased when more addition of titanium carbide particles.

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Table 3. Experimental observations for drilling of niobium metal matrix.

| S. No | A: Cutting speed (m min$^{-1}$) | B: Laser power (W) | C: Gas pressure (bar) | MRR (gm min$^{-1}$) | SR ($\mu$m) |
|-------|---------------------------------|--------------------|----------------------|---------------------|------------|
| 1     | 1                               | 1200               | 15                   | 3.21                | 2.5        |
| 2     | 2                               | 1000               | 20                   | 4.21                | 3.2        |
| 3     | 1.5                             | 1000               | 15                   | 6.25                | 3.9        |
| 4     | 1                               | 1000               | 20                   | 2.14                | 4.2        |
| 5     | 1                               | 800                | 15                   | 3.32                | 6.3        |
| 6     | 2                               | 1200               | 15                   | 7.28                | 2.2        |
| 7     | 2                               | 800                | 15                   | 3.68                | 6.9        |
| 8     | 1.5                             | 1200               | 10                   | 4.92                | 1.9        |
| 9     | 1.5                             | 800                | 10                   | 8.21                | 5.9        |
| 10    | 1.5                             | 1200               | 20                   | 8.25                | 2.3        |
| 11    | 1.5                             | 1000               | 15                   | 4.28                | 3.3        |
| 12    | 1.5                             | 1000               | 15                   | 4.1                 | 3          |
| 13    | 2                               | 1000               | 10                   | 5.97                | 4.6        |
| 14    | 1                               | 1000               | 10                   | 4.98                | 2.6        |
| 15    | 1.5                             | 800                | 20                   | 2.29                | 7.1        |

Table 4. Analysis of variance for MRR.

| Source            | Sum of DF | Mean | F-value | p-value | Sum of DF | Mean | F-value | p-value |
|-------------------|-----------|------|---------|---------|-----------|------|---------|---------|
| Model             | 49.90     | 9    | 5.54    | 0.019   | 43.47     | 9    | 4.83    | 0.0006  |
| A: Cutting speed  | 7.01      | 1    | 7.01    | 0.0271  | 0.21      | 1    | 0.21    | 1.43    |
| B: Laser power    | 4.74      | 1    | 4.74    | 0.051   | 37.41     | 1    | 37.41   | 0.782   |
| C: Gas pressure   | 6.46      | 1    | 6.46    | 0.031   | 0.40      | 1    | 0.40    | 2.74    |
| AB                | 3.44      | 1    | 3.44    | 0.082   | 0.20      | 1    | 0.20    | 1.37    |
| AC                | 0.29      | 1    | 0.29    | 0.556   | 2.25      | 1    | 2.25    | 15.25   |
| BC                | 21.39     | 1    | 21.39   | 0.002   | 0.16      | 1    | 0.16    | 1.08    |
| AA$^2$            | 4.05      | 1    | 4.055   | 0.065   | 0.16      | 1    | 0.16    | 1.13    |
| BA$^2$            | 1.09      | 1    | 1.09    | 0.276   | 2.74      | 1    | 2.74    | 18.62   |
| CA$^2$            | 0.91      | 1    | 0.91    | 0.315   | 0.005     | 1    | 0.005   | 0.03    |
| Residual          | 3.66      | 5    | 0.73    |         | 0.73      | 5    | 0.14    |         |
| Lack of Fit       | 0.82      | 3    | 0.27    | 0.893   | 0.3175    | 3    | 0.10    | 0.50    |
| Pure Error        | 2.84      | 2    | 1.42    |         | 0.42      | 2    | 0.21    |         |
| Cor. Total        | 53.57     | 14   |         |         | 44.20     | 14   |         |         |

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The morphology, material characterization and composition of the niobium metal matrix were analyzed through SEM and EDAX. Titanium carbide particles were providing good bonding strength between the alloying elements.

The metal matrix was synthesized through sintering process and machined by unconventional machining processes such as ultra sonic and laser beam machining processes.

The power rating was the most influential parameter which affects the material removal rate and percentage of slurry concentration which affect on surface finish.

The optimal parameters of MRR and SR were found by taguchi approach. The maximum material removal rate was attained at power rating (300 W), percentage of slurry concentration (30%) and grit size (400). The minimum surface roughness was attained at power rating (200 W), percentage of slurry concentration (40%) and grit size (300).

From analysis of variance, cutting speed and gas pressure are found to be significant parameters for MRR and Sr

ORCID iDs

G Sai Krishnan @ https://orcid.org/0000-0001-7828-8399

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