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Surface and Wear Investigation on Microwave Sintered Nitinol Composite

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The present research of the article is used to describe the microwave sintering of nitinol composite and its surface topography analysis by atomic microscopy (AFM). Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) technique is used to analyze the characterization and alloying composition of the composite. Silicon carbide is the reinforcing particle between nickel and titanium. Wear test is conducted to investigate the worn surface morphology of the sintered specimen. The synthesized composite has exceptional material properties such as ultimate tensile strength of 1020MPa, 16% of elongation to fracture, 7.51 g/cc of density and hardness of 310 HV. Low wear rate of 0.01 mg/m is perceived at minimum load and minimum sintering time. Surface roughness is varied from 22.87 to 54.05nm at different sintering time. Maximum roughness height of 327.6 nm is observed in the surface profile of the composite.

Keywords*: Nitinol composite, Characterization, Microwave sintering, Surface topography, Silicon carbide, Wear.*

1. Introduction

The application of the nitinol is rapidly increasing in medical instruments, electrical and electronics components. Nitinol alloy has different crystal structure of nickel and titanium. It has 6.45g/cc of density and 15% of elongation to fracture. Due to various crystal structures, it has outstanding physical and mechanical properties. The range of the ultimate tensile strength between 754MPa and 960Mpa is observed for nitinol alloy. The fabrication technique, material characterization and applications of the nitinol alloy have been studied. The effect of heat treatment and its variation of microstructure have been investigated with various temperature conditions[1]. Thermo mechanical behavior and microstructure of nitinol alloy was investigated with different parameters. The application of nitinol actuator springs was studied[2]. The morphology, grain growth and phase transformation of the nitinol alloy was studied. The crystallographic texture was analyzed with different macroscopic response[3]. Tensile behavior, grain orientation and interfacial strength between nickel and titanium were analyzed by different level of strain[4]. The effect of different parameter on strength, microstructure and surface topography was investigated in nitinol alloy[5]. Analysis of micro-hardness, formation of crystalline structure and accumulation of strain energy was analyzed in nitinol alloy. The material properties of nitinol alloy and its quality characteristics were compared with other alloys[6,7]. Wear and mechanical behavior of the nickel titanium alloy was depends on the grain size of the particles. Hardness and wear rate of nickel titanium alloy has been improved by multi directional forging method[8,9]. Wear resistance of the titanium nickel alloy has been improved by high heat resistance and fracture toughness under different sliding velocities[10]. Tribological characteristics and morphology of the worn surface were analyzed by SEM images of the nickel alloy at elevated temperature. The chemical composition analysis was conducted on the wear worn surfaces[11]. The quality characteristics of wear were analyzed in titanium based bearing surface. The coefficient of friction was depending on the formation of oxide layer on the surface[12]. The microstructure and dry sliding wear performance of the titanium composite was analyzed under different temperatures. Wear rate was reduced and coefficient of friction has been increased due to the presence of titanium oxides[13].

The present research of the article is used to analyze the microwave sintering of silicon carbide based nitinol composite. The surface topography of microwave sintered specimen was analyzed by AFM. The material properties, characterization, alloying composition and wear behaviors were studied.

2. Experimental Details and Methodology

Figure 1 represents the stages were followed to convert powdered particles in to sintered specimen. The various factors

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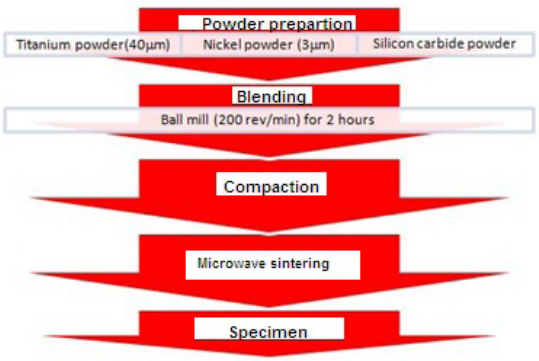

Figure 1. Stages of sintering process.

such as temperature, frequency of radiation, chemical state of the material, size of the particles and heating mechanism are affecting the microwave sintering process. In the powder preparation, titanium, nickel, and silicon carbide particles were considered. Blending is the process used to mix the particles in homogeneous stage. In the compaction stage, the powdered particles were renovated in to green samples. In the microwave sintering process, the heating process was involved to make the sintered specimen[14].

After preparing the specimen, it undergoes microscopic investigation to examine the surface morphology. In the experimental investigation, nickel and titanium powder particle was combined with silicon carbide. Silicon carbide with 1.5 weight percentage was considered for this experimental investigation. It has high strength and thermal conductivity. It has excellent resistance against oxidation, temperature, chemical and hardness. More importance was given for blending process to achieve uniform distribution particles in sintered specimen. Planetary mill was rotated at 200 rev/min for 2 hours to mix the powders with the ball to powder ratio of 4:1. Then the pounded particles are compacted using cold pressing process with a pressure of 22.54 MPa stated in to produce green samples[15]. In this investigation, two samples are prepared with size of diameter 15mm and 6 mm height. After that the green samples are followed with micro wave sintering with different sintering time of 5 minutes and 30 minutes with 1000^0C. Schematic layout of micro wave sintering setup was neatly presented in Figure 2. The microwave radiations (2.5 GHz) are generated with the help of two magnetrons activated at 4.5 kW. The green samples were put inside the alumina crucible. Green samples were fully converted in to sintered samples with the help of micro wave heating rate at 20-30^0C/min. Infrared pyrometer was attached externally to sense the sintering temperature during process. Argon gas was used to avert oxidation of Nitinol composite. The total setup was designed to rotate and after producing the sintered samples, round specimens was converted in to square specimens in the dimension of 4 mm x 4 mm and height of 8mm to explore the effect microscopic behavior. Specimen was fabricated with the face and ending diameter of 10 mm, neck diameter dimension of 6mm with a length of 30mm, as per the ASTM E8M11 standard for tensile test (FIE-UTM-40 Ton) . Density of the sintered specimen was evaluated by using Archimedes principle.

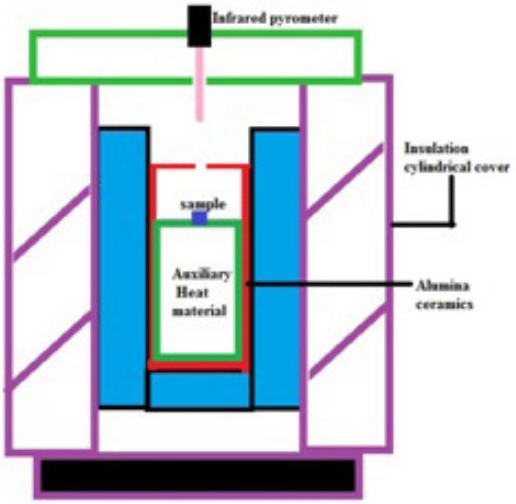

Figure 2. Schematic layout of the microwave sintering setup.

Table 1. Material properties of Nitinol composite.

Material properties	Value
Tensile strength	1020MPa
Hardness	310HV
Elongation to fracture	16%
Density	7.51

Vickers hardness tester (ALPHA 400 VH) was successfully implemented to compute the hardness of the specimen. The material properties were listed in the Table 1.

3. Result and Discussion

3.1. Microscopic analysis

Figure 3 shows that the SEM image of titanium powder with the size of 40 μm and nickel powder with 3μm was used to manufacture the nitinol composite using microwave sintering method. Figure 4 illustrates the alloying composition of microwave sintered specimen of nitinol composite. From EDS figure, it was clearly observed that the composite contains nickel with 49.5%, titanium with 49% and SiC with 1.5%. XRD analysis was clearly shown in Figure 5, it was analyzed to distinguish the phase composition of the sintered sample. SEM image was used to evaluate the microscopic behavior of the composite. In this examination, two sintered samples were prepared with two different methods. First method was used to fabricate the specimen with the sintering temperature of 1000^0C and sintering time of 5 minutes. Second method is to prepare the specimen with sintering temperature of 1000^0 C and sintering time of 30 minutes. After fabricating the specimen, it was finely polished with diamond paste to attain mirror finished specimen. Then it was tested to analyze the microscopic behavior by using FESEM. SEM images of the specimen with sintering temperature of 1000^0C with sintering time of 5minutes and same temperature with 15 minutes was clearly presented in Figure 6a and (b) respectively. Porosity explains the percentage of volume of

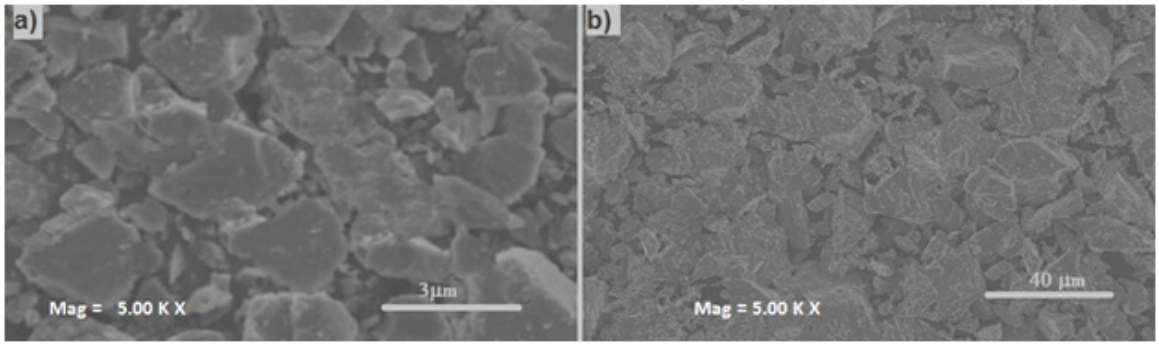

Figure 3. SEM image of (a) Ti and (b) Ni Powders.

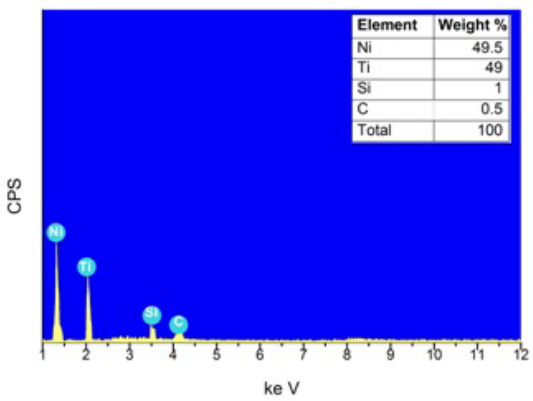

Figure 4. EDS for the microwave Nitinol sintered sample.

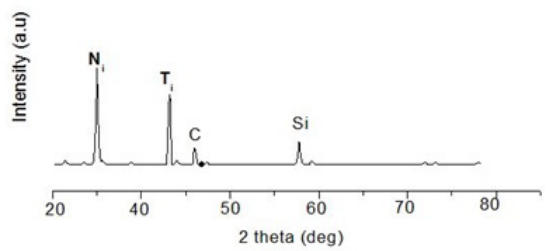

Figure 5. XRD analysis of the sintered composite.

void or cavity formation during the sintering process. It mainly depends upon the sintering temperature and sintering time. The matrix particles were clearly observed in SEM images. Due to heat removal and sudden cooling, different crystals were observed[16]. It was obviously noticed from the Figure 6, the surface contains of cracks, steps and stairs because of surface oxidation effect[17,18]. Open pores were clearly visible in the Figure 5. Owing to more inherent diffusion coefficient, vacancy formed in diffusion zones, resulted in formation of high porosity between the titanium and nickel powder mixed together[19].The pore feature of nitinol composite was shown in Figure 6a and 6b. In Figure 3a and 3b diameter of the pores were deeply analyzed with respect to sintering time and it was noted that the inner surface of the pore was coarse at minimum sintering time and diameter is drastically increased with an increase of sintering time. Thermal energy is a key factor for pore formation. Temperature difference and sintering compaction pressure is also causes the formation of pores. Nickel combined with titanium particles entrenched on the surface[20] and it was noted in the same Figure 6a and 6b.In deep investigation of microscopic approach of nitinol, pores with irregular size and shapes was formed in the surface clearly presented in Figure 6a and 6b. In this investigation, the sintering time plays a most vital role in pores formation. Compared with Figure 6a and 6b, pore size is larger in Figure 6b only because of overheating of the particles for sintering time of 30 minutes. Pore formation was occurred due to Ti atoms diffused in Ni regions owing to imbalance mass transfer was clearly investigated. Spherical pores are also noted in Figure 6a due to shortest sintering time.

3.2. Surface topography

Surface texture of the sintered nitinol composite was clearly presented in Figure 7 and Figure 8 with sintering temperature of 1000^0C at 5 minutes and 30 minutes respectively. Atomic force microscope was representing the three dimensional surface textured model (Figure 7). It was observed with spikes and valleys profile that denotes the surface texture has rough surface profile. Dark spots are also observed in pores due to innate diffusion coefficient[21]. From AFM image (Figure 8), it was obviously observed with more flat irregular shapes which denote irregular shape of coarse pores due to sintering time for 30 minutes. More sintering time and overheating of particles create the surface profile with irregular massive pores.

From Figure 9 and Figure 10, it was shown the surface morphology of the sintered Nitinol composite for sintering time of 5 minutes and 30 minutes respectively. By using the line A and B in the profile, it was deeply investigated of the surface profile using micro wave sintering process. It was interesting to know the importance of the surface factors like average roughness, root mean square, maximum height of the roughness, maximum peak to valley roughness, skewness and kurtosis. The skewness and kurtosis parameters were plays a more imperative role to identify whether the surface is smooth or rough. While comparing both the surface profile maximum surface roughness value (54.05 nm)was observed in the sintered specimen for sintering time of 30 minutes (Figure 8). The maximum valley depth was also found

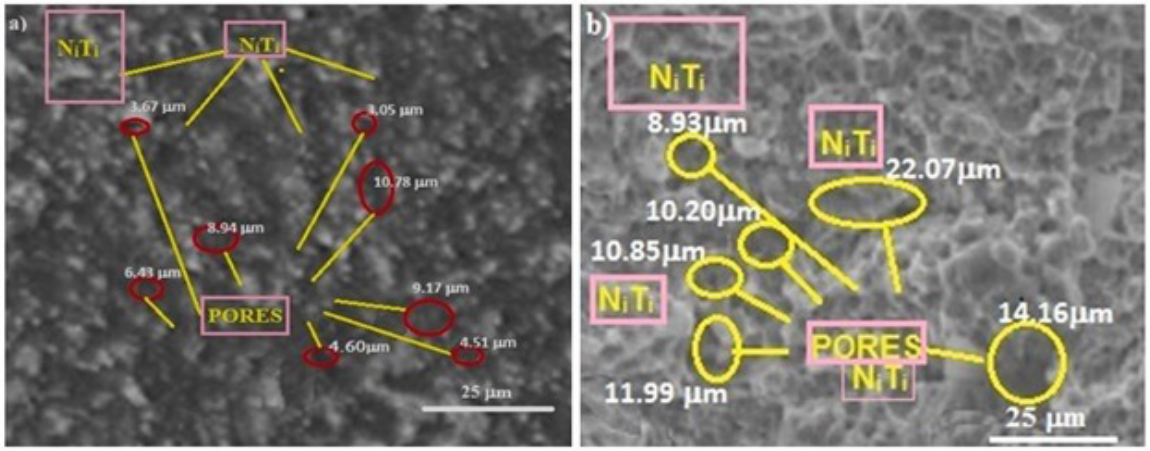

Figure 6. SEM images of Nitinol composite at 1000^{0}C temperature with a) sintering time for 5 min b) sintering time for 30 min.

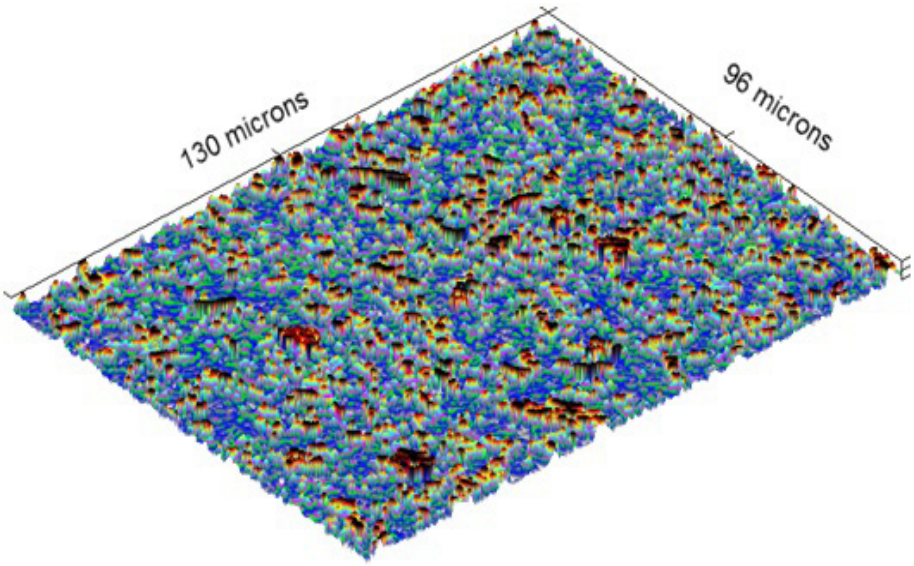

Figure 7. AFM image of Nitinol composite at sintering temperature 1000^{0}C for 5 minutes.

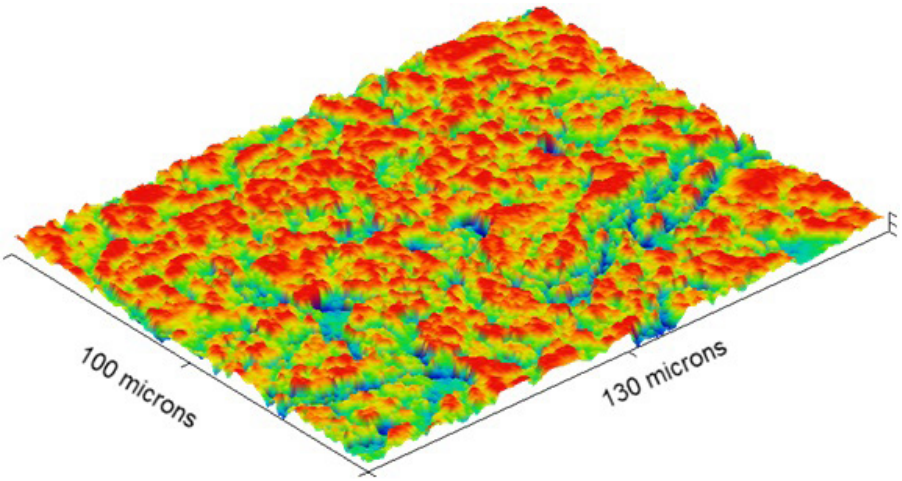

Figure 8. AFM image of Nitinol composite at sintering temperature 1000^{0}C for 30 minutes.

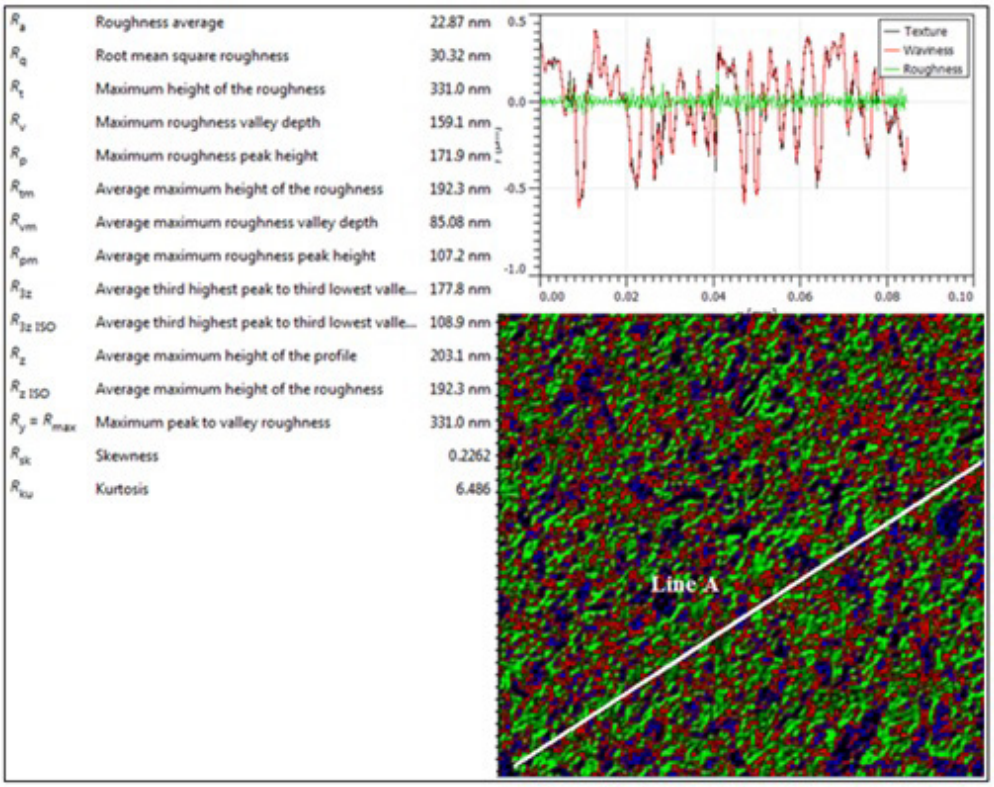

Figure 9. Surface parameters of Nitinol composite at 1000^{0}C for 5 minutes.

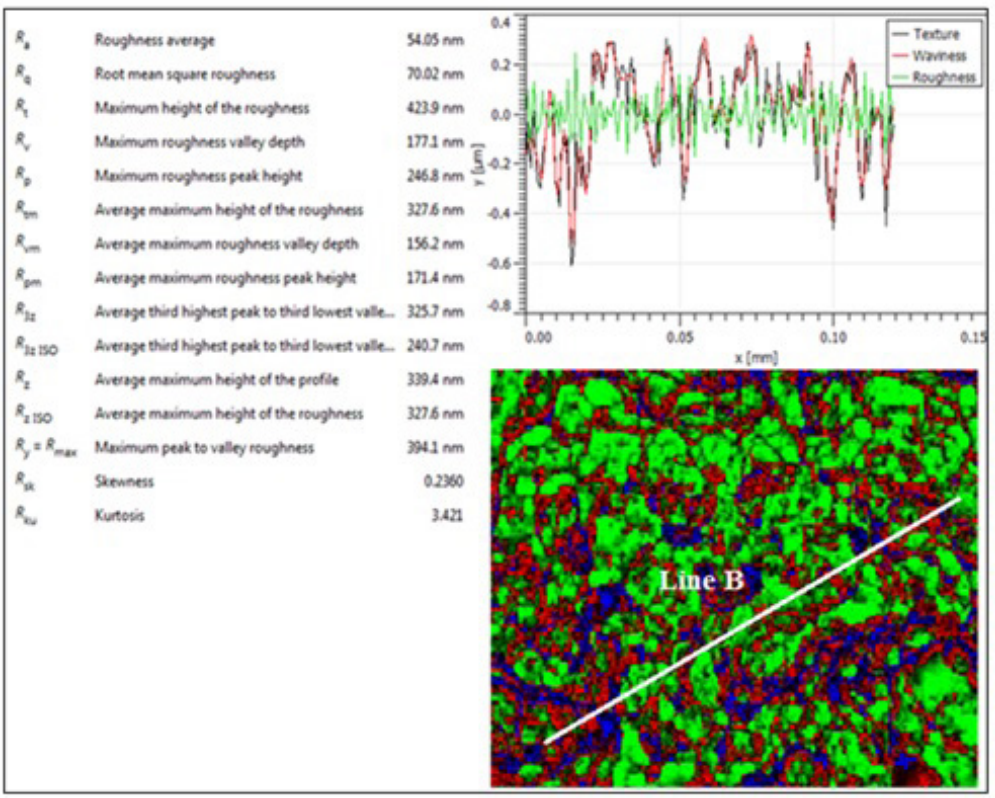

Figure 10. Surface parameters of Nitinol composite at 1000^{0}C for 30 minutes.

in the specimen represented in Figure 10 with a value of 177.1 nm. The kurtosis value is more than 3 indicates the surface has high spike profile and if the value is lesser than 3, it seems the surface profile is flat. From surface parameter profile (Figure 9), the value of 6.486 was observed for kurtosis. Hence, it was considered as spike profile. But in the Figure 10 the value of the kurtosis is 3.421 so the profile appears with less spike profile compared with Figure 9. The skewness value for both the specimen represented in Figure 9 and Figure 10 is lesser than 3, so that the profile slightly flat not like that hill profile[22].

4. Wear Test Analysis

The different factors such as composition of the material, size of reinforcement particles, material structure, physical and mechanical properties are used to affect the wear behavior of the composite. Pin-on-disc tribometer was involved to conduct wear test on Nitinol composite. As per ASTM standard, wear testing specimens[23] were prepared with the diameter of 35 mm and 8 mm thickness. Average surface roughness of 0.4 μm was maintained for the specimen. In the wear test analysis SAE52100 bearing steel with the hardness value of 58 HRC was act as a pin[24]. The wear test was planned to conduct with the load of 20, 40 and 60 N with the varying sliding velocity from 1 to 3 m/s and sliding distance from 500m to 1500m.The worn out surface of the specimens were accurately characterized by using SEM analysis.

The wear rate of Nitinol composite with varying load condition from 20N to 60N was shown in Figure 11. From the figure, it was clearly shown that the variation of wear rate at different sintering time. Based on previous research study, the sliding distance of 1000m suggested for wear analysis of nitinol alloy[25]. Wear rate is significantly reduced with further increase of load condition at 60N. Figure 12a and 12b illustrate the wear rate of Nitinol composite with varying sliding distance at maximum and minimum sintering time respectively. In this figure, it was clearly reported that the wear rate was gradually decreased with an increase of sliding distance from 500m to 1500m. Little amount of wear rate deviation was observed at the sliding distance of 1500m, for the normal load condition of 20, 40, and 60N. Minimum wear rate was noticed at the normal load conditions of 20N and 40N.Wear rate has been decreased with increasing of sliding distance and it was noticed from Figure 11a and 11b. It was concluded that sliding distance was indirectly proportional to the wear rate for both maximum and minimum sintering time.

Figure 13 shows the SEM image of Nitinol composite after the wear test analysis. Figure 12a illustrate the SEM image of the sintered specimen prepared at minimum sintering time and (b) represented the SEM image of the specimen prepared at maximum sintering temperature in microwave sintering process. Owing to cruel adhesive wear takes place during wear test, metallic wear debris was developed and it was noticed in Figure 13a and 13b. Due to metal to metal contact at the maximum sliding speed and load condition,

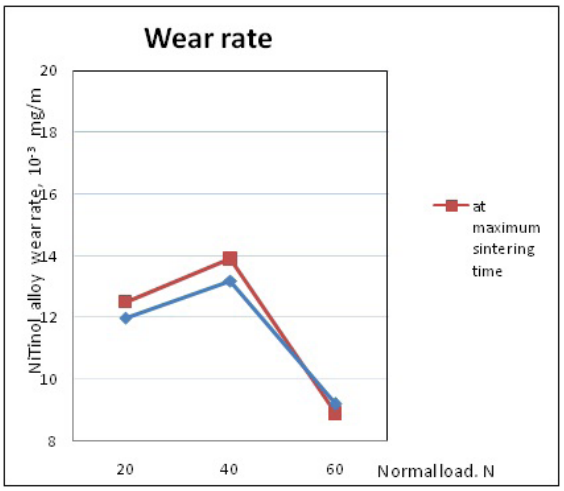

Figure 11. Wear rate of Nitinol composite with varying normal load at the sliding distance of 1000m.

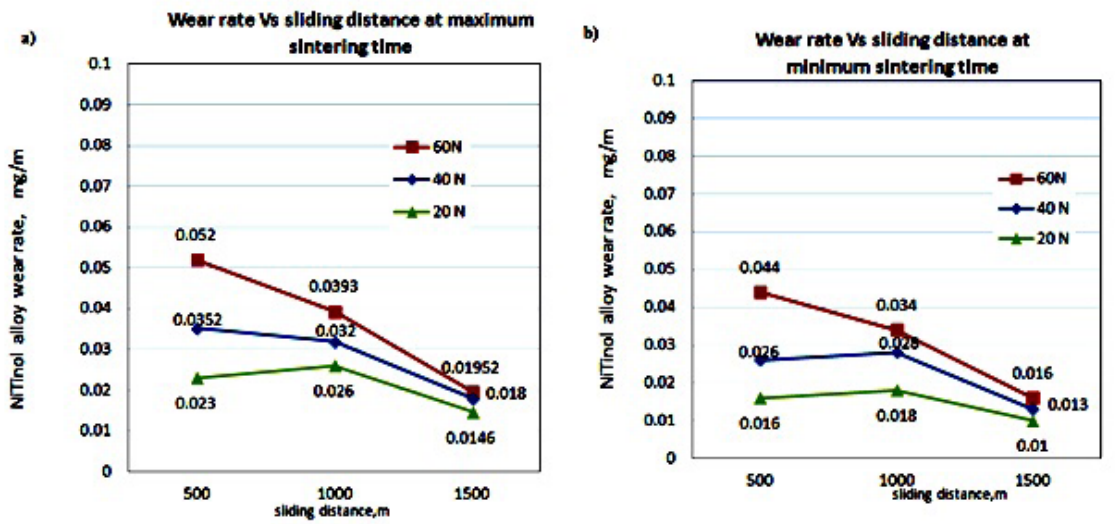

Figure 12. Wear rate of Nitinol composite with varying sliding distance a) at maximum sintering time b) at minimum sintering time.

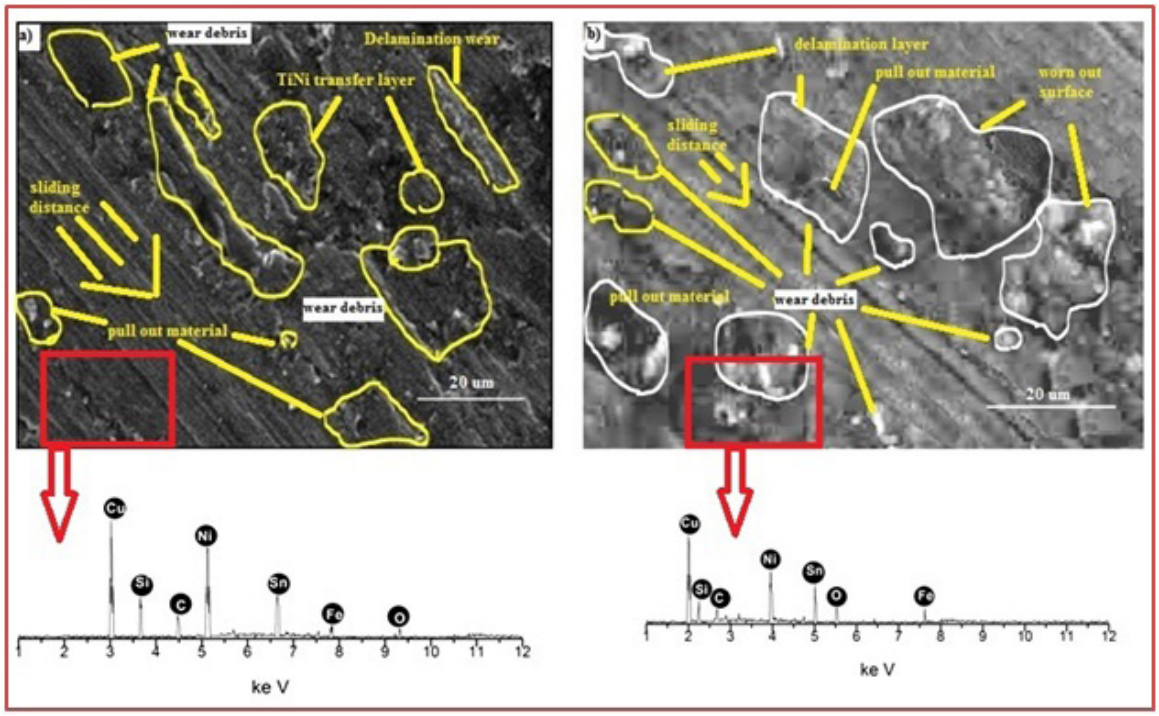

Figure 13. SEM micrograph of worn surface of Nitinol composite a) prepared at minimum sintering time b) prepared at maximum sintering time.

metal fracture and worn out surface was noticed[26].At higher load condition, crack propagation creates over the surfaces and shown in Figure 13, due to sliding action in between Nitinol composite and SAE52100 bearing steel during the wear test. Delamination of the layer was found in the surfaces of the sintered specimens in Figure 13a and 13b, due to deep plastic deformation[27].Due to abrasive action between the contact surfaces in wear analysis, Ti-Ni transfer layer was found in Figure 13a and 13b. The maximum wear rate was occurred at maximum load of 60 N. The micro-ploughing action was created in between the contact surfaces at minimum sliding distance and higher load conditions may be induced pullout material and worn surfaces[28,29].

5. Conclusion

The following points were added in conclusion based on the experimental result of microwave sintered nitinol composite.

- The different stages of microwave sintering process were investigated for synthesis of nitinol composite.
- The synthesized nitinol composite has ultimate tensile strength of 1020MPa, 16% of elongation to fracture, 7.51 g/cc of density and hardness of 310 HV.
- The characterization and alloying composition of nitinol composite was analyzed with the aid of SEM, XRD and EDS techniques.
- Pore formation was deeply investigated through the SEM image of the sintered composite at both the minimum and maximum sintering time
- Pore size variation found minimum value in between 3.67 micron to 10.78 microns at minimum sintering time conditions, very large pore size of 22.07 micron was noticed in the specimen fabricated at maximum sintering time
- Wear test analysis was effectively conducted on the various wear patterns and worn surfaces was analyzed through SEM images.
- In both minimum and maximum sintering time, sliding distance was in indirectly proportional to the wear rate.
- Maximum wear rate of 0.052 mg/m was observed at maximum sintering time. Minimum wear rate of 0.01 mg/m was perceived at low load condition and minimum sintering time.
- The surface topography was implemented and to examine the surface of the worn profile using AFM.
- Surface roughness was varied from 22.87 to 54.05nm at sintering time of 5 to 30 minutes.
- Maximum roughness height of 327.6 nm was found at maximum sintering time of 30 minutes.
- Skewness and kurtosis parameter was considered as the most important parameter to define the profile of the worn surface.

6. References

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