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High temperature friction and wear experimental studies on 3D printed nickel iron base superalloy

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Abstract: In this paper, a nickel-base alloy is developed through 3D printing process following the laser sintering technique for high temperature wear application. The mechanical properties such as hardness, strength and density are evaluated to just the quality of 3D printed material. The Vickers hardness of the 3D printed nickel alloy is 265 Hv which is very close to the commercial alloy available superalloy in the market. The density of the 3D printed nickel alloy is 8.2 g/cc and for the conventional superalloy is 8.17 g/cc. From the sliding wear analysis, it has to be noticed that with respect to increase in load the coefficient of friction and wear rate found increasing gradually throughout the test duration. This is due to the frictional force and the working environment applied during the investigation. Mass loss is noticed at 10 N-150°C; to maximum of 0.306 g. The influence of thermal and mechanical load has influenced the contact surface damage.

Keywords: 3D printing; nickel alloy; wear; temperature; surface topography.

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1 Introduction

The application of nickel alloys in aero engine is in high demand due to the mechanical properties and metallurgical behaviour at different working conditions. Under severe operating condition, the materials are prone to failure with mechanical loading and aggressive working environment. The early failure of the component is due to the worn and fatigue conditions (Xu et al., 2021). In general, the nickel alloys do undergo high abrasive wear under mechanical loading condition. Literatures are available to discuss about the nickel-based superalloy and its mechanical failure (Birol, 2010; Chakraborty et al., 2021). In a combination mechanical loading and sliding friction, the materials are vulnerable towards surface damage in the form of wear. In order to protect the surface of the material from the damage coatings and heat treatment plans are proposed to increase the life of the component (Derelizade et al., 2022; Khan et al., 2017).

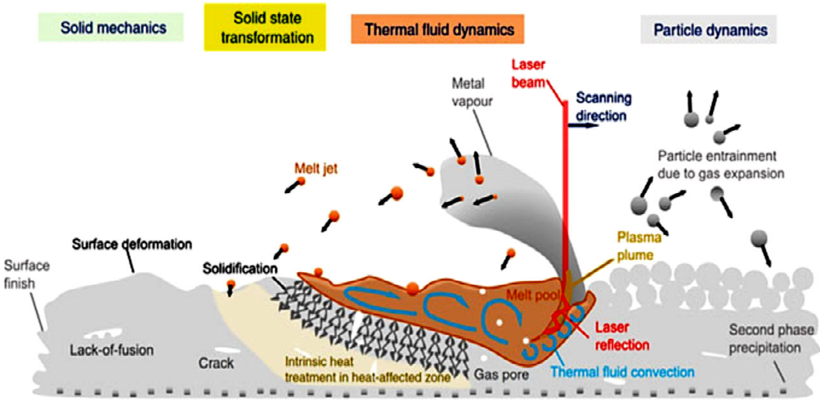
In addition to mechanical loading, increase in temperature leads to phase transition and surface oxide formation (Jiang et al., 1994). As the nickel alloys are highly sensitive towards working temperature and the rapid phase transformation starts around 500°C for a minimum of soaking at 30 minutes (Jambor et al., 2017). In consideration with metallurgical phase transformation of nickel-iron base superalloy, the experimentations are planned and performed below 400°C and the precipitate formation of niobium is controlled. In addition, there might be some possibilities on surface oxide formation with increase in temperature. There is some ideology developed by the researcher that to replace the issues in conventional superalloy, additive built alloys are in plan for the future research scope. The 3D printing process are carried out with different technology similar to welding (as weld arc additive manufacturing) and powder sintering (powder metal laser sintering) processes (Bhaduri et al., 2004; Chakraborty et al., 2021). Selection of fretting wear and its working process conditions are still in complex to fix for nickel iron base superalloy (Samuel et al., 2020). It has to be admitted that, from the literature the conventionally developed nickel iron base alloys are widely used for fretting wear and the sliding wear studies. Especially, the power shaft is made of nickel it is supported in the hub for better energy transmission within the turbine hot section. The contact surface causes wear and tear due to the sliding friction and applied load on the power. In order to counter the working environment material development and the engine life improvement has become a challenge for researchers. As a lead, the development of superalloy through 3D printing and fretting wear analysis for turbine application is open to researcher.

As a research gap, the development of nickel alloy is a great challenge for past two decades. Superalloys are stable at ambient working environment and the metallurgical transition occurs on increase in temperature. The alloys developed are highly sensible towards increased working environment. To sustain the mechanical and metallurgical properties of the superalloy, additive manufacturing process is adopted. In this paper a nickel iron base superalloy is proposed to develop through 3D printing process using laser powder bed fusion method. The developed alloy is used to study the mechanical properties to substantiate the equivalence with conventional nickel iron base alloy which is commercially available in the market. Further the friction and wear analysis on 3D printed nickel iron base alloy is subjected to friction and wear analysis under high temperature condition for different loading conditions. The outcome of the research will support young researcher to identify the solution and foundation on application of nickel iron base alloy for friction and wear application at high temperature.

2 Experimental procedure

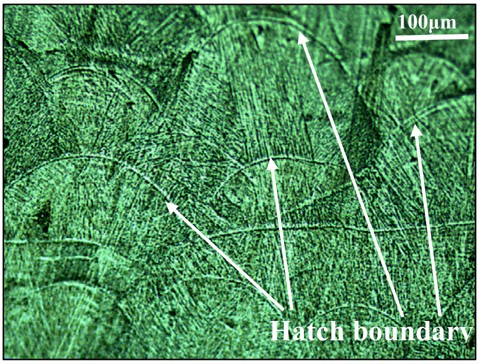
A nickel iron base superalloy (Inconel 718 grade) with a composition of; Ni – 52.3%, Fe – 19.8%, Cr – 17.5%, Nb – 4.7%, Mo – 3.38%, Ti – 1.1% and traces of Al, Si, C, Co and W are developed through 3D printing (additive manufacturing) process from a standard atomised powder 30–50 µm. The chemical composition of the alloy is confirmed through the spectroscopic analysis. The test samples in the form of square pieces (10 × 10 mm) are built with EOS M280 model direct metal laser sintering machine. The standard process conditions as follows: laser energy – 285 w, laser scan speed – 950 mm/s, hatching – 0.15 mm in horizontal direction and hatch thickness – 40 µm (Raj et al., 2022). The use of laser source for metal 3D printing is as given in the Figure 1. The metal powders are scanned with laser beam and the localised fusion are made directly followed by rapid solidification process. During this high-speed laser beam scanning on the metal powder, different forms of transformation phases involved as; metal powder → metal pool (thermal fluid) → solid state transformation → solid mechanics and finally a solid metal is developed (Khan and Jappes, 2022). At this stage, the materials build through direct metal laser sintering (DMLS) process are in anisotropic properties which are highly induced due to rapid fusion and solidification process the metal powders. Figure 2 shows the optical microstructure of 3D printed nickel iron base superalloy developed through laser sintering process. It has clear structure to reveal the layer-by-layer of metal added and the hatch geometry. Dendrite structure with the hatch direction shows the rapidly solidified metal following solid mechanics during printing. The basic mechanical properties of the 3D printed nickel iron base superalloy are evaluated to compare the developed material with conventional material following the standards with designation of ASTM E8. Further the samples are printed to a cubic shape of 8 mm side for the test, considering the economics of 3D printed material cost. Further the cubic sample printed for the investigation are brazed with austenitic stainless steel (SS316L) pin of 8 mm in diameter to maintain the reliable working condition for high temperature friction and wear analysis. Figure 3 shows the photo image of the 3D printed nickel iron base superalloy brazed with SS316L pin for experimental investigation.

Figure 1 Schematic illustration for laser processed metal 3D printing and phase transformation (see online version for colours)



Source: Khan and Jappes (2022)

Figure 2 Microstructure of 3D printed nickel iron base superalloy developed through laser metal sintering processing (see online version for colours)



The friction and wear analysis on the 3D printed nickel iron base superalloy is conducted on pin-on-disc sliding wear test rig. The Photo image and working model of the friction and wear test setup is given in the Figure 4. The experiments are performed following the internationally recommended standard procedure designated as ASTM G99-05 using the pin-on-disc tribometer (model: Ducom – TR20LE) (Samuel et al., 2020). The test specimen height should be 30 mm and the pin diameter has been taken as 8 mm. The disc is made of high-grade hard steel which is considered for the application of turbine power shaft made of nickel alloy fixed over the hard steel hub to control friction vibration and for energy transformation. The instrument is integrated with a data acquisition system to record the physical changes such as sliding wear (in μm) and frictional force (in N) involved during the sliding wear analysis.

Figure 3 Photo image of 3D printed Ni alloy brazed on stainless steel pin (see online version for colours)

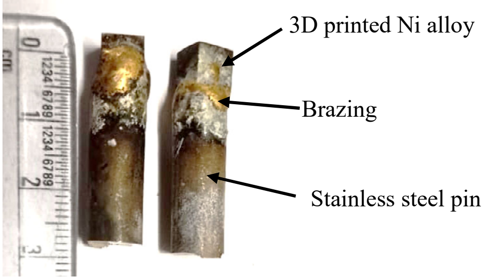
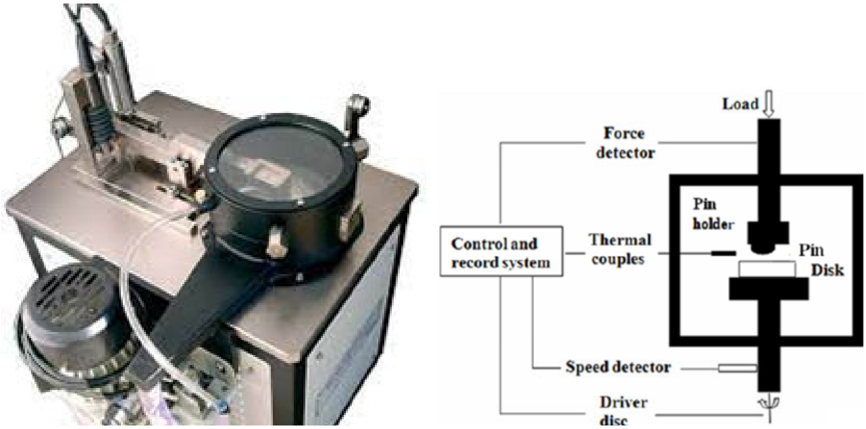


Figure 4 High temperature pin on disc wear test rig and working model (see online version for colours)



The experiments are planned to conducted for nine different combination of process parameters as given in the Table 1. The experimental design is made with three different factors; sliding velocity (m/s), applied load (N) and temperature (°C) at different combination. During investigation, the wear track diameter is fixed as 80 mm and the sliding distance is maintained constant for a distance of 500 m.

Table 1 Experimental plan to conduct friction and wear analysis at high temperature

Trial no.	*Load (N)*	*Temperature (°C)*
1	5	100
2	5	150
3	5	200
4	10	100
5	10	150
6	10	200
7	15	100
8	15	150
9	15	200

The physical changes in terms of wear (μm) and the frictional force (recorded value in N) are taken as a response to study the friction and wear behaviour of 3D printed nickel iron alloy. Subsequently, the mass changes in 3D printed sample (before and after wear analysis) are also recorded for investigation. Following mathematical relations are used to study the behaviour of 3D printed material for friction and wear analysis.

$$Volume\ loss = \frac{mass\ change}{density} \times 1{,}000\ [\text{mm}^3] \tag{1}$$

$$Wear\ rate = \frac{volume\ loss}{applied\ load \times sliding\ distance}\left[\frac{\text{mm}^3}{\text{Nm}}\right] \tag{2}$$

$$Wear\ resistance = \frac{1}{wear\ rate}\left[\frac{\text{Nm}}{\text{mm}^3}\right] \tag{3}$$

Using the above expression, the mathematical values are predicted to infer the properties of sliding wear behaviour of 3D printed nickel iron base alloy at high temperature. Further the tested samples are investigated with scanning electron microscope to infer the wear mechanism and surface topography with respect to the mechanical friction induced during the experimentation.

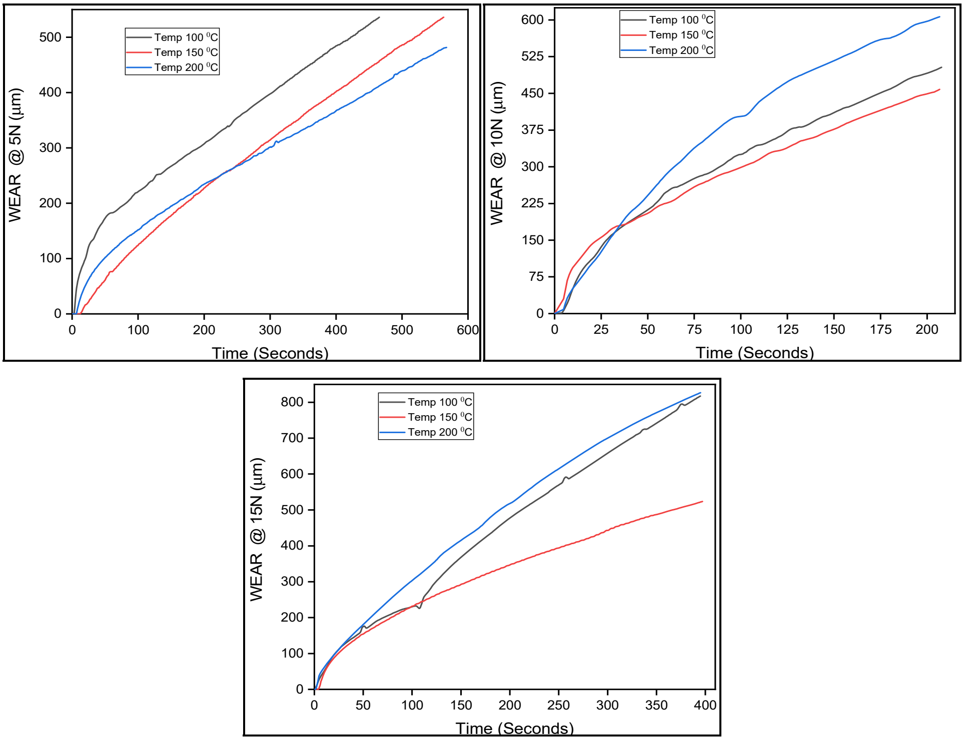
3 Results on experimental analysis

3.1 Wear and coefficient of friction

The 3D printed nickel iron base superalloy developed through laser powder metal sintering has been evaluated for basic mechanical properties as given in the Table 2. The samples are metallurgically polished and the mirror surface is used to measure the Vickers hardness (weight of 300 g for 10 s). The measured hardness is 265.5 Hv for 3D printed nickel iron base alloy which is very close to conventional superalloy (278 Hv) of same grade material. The slight changes in hardness might be due to the delta phase developed during as printed stage. It indicates that the delta phase formed due to rapid solidification has influenced to produce the range of hardness in the as printed material (Raj et al., 2021; Raghavulu et al., 2022). Just like the case, the density of the 3D printed alloy has 8.2 g/cc and conventional alloy has 8.17 g/cc. At the same, the mechanical load test shows 1,195 MPa for 3D printed and the 1,240 MPa for conventional alloy. Obviously, the density of dendrite is slightly weighing more than the homogenise material as reported in the literatures. The microstructural of the 3D printed material observed through the optical microscope (Figure 2) confirms the presence of dendrite structure throughout the bulk developed through 3D printing process.

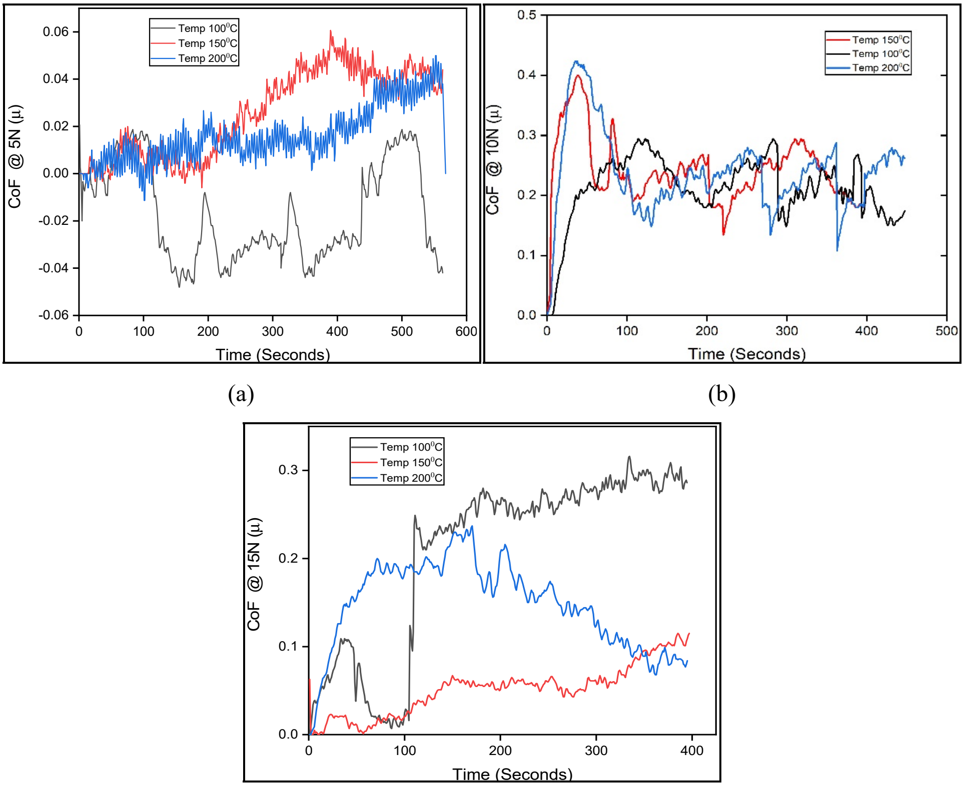
Experimentation continued with 3D printed nickel iron base alloy with high temperature pin on disc test rig. The results recorded during the experimentation are discussed in detail with significant justification on the outcome of the investigation. Testing is performed in a unidirectional sliding wear concept to study the behaviour of 3D printed nickel iron base alloy; with depth of wear in pin (in μm) and coefficient of friction (in μ) generated during sliding resistance over the counter material. The results are inferred with respect to applied load such as 5 N, 10 N and 15 N for the sliding wear and coefficient of friction for the temperature 100°C, 150°C and 200°C working condition. Figure 5 indicates the difference in wear recorded for different applied load and working temperature through the data acquisition system interconnected with the test rig. The pin material found with increase in wears with respect to increase in applied load condition. At 5 N applied load condition, the minimum wear of 461 μm is recorded for 200°C at the end of experimentation. Initially the wear for all the three conditions found increasing in a steep up to 200 μm for about 50 s to 100 s and coined towards a track with gradual lead. Similarly, the wear found uniform increasing to 150 μm for 20 s to 45 s at 10 N and wear raised to maximum of 609 μm at 200°C. For 15 N, up to 50 s the wear is equal for all the conditions and increased drastically with respect to the temperature to a maximum of 802 μm at 200°C. It has been compared with the existing literature and noticed that the 3D printed material has high wear resistance as equal to thermal barrier coatings (Khan et al., 2017). The temperature and applied load have highly influenced towards the material damage in the form of wear. The failure is due to the resistance of materials towards friction and the changes in friction are recorded as given in Figure 6.

Figure 5 Wear measured on the 3D built nickel alloy pin material at different applied load and working temperature with respect to time (see online version for colours)



The coefficient of friction for the proposed test material is recorded and plotted with respect to the applied load and working temperature. Figure 6 shows the variation in coefficient of friction for different applied load and working temperature. For minimum load of 5 N there was drastic fluctuation in friction reached to a maximum of 0.4 μ and settled between 0.2 μ to 0.3 μ due to frictional force generated. The distraction in coefficient of friction at 5 N is because of the applied load. At minimum load, the sliding properties are fluctuating and indicating the noise over the record. Subsequently for the maximum applied load of 10 N the frictional force gone stabilised and the co-friction is stable. There is some notice with variation in coefficient of friction at 15 N [Figure 6(c)]. With increase in temperature, the material developed with nickel base might be sensitised and influenced towards frictional wear. This might be due to the distraction in contact area in the form of abrasive wear. It is directly correlated to the difference in mass loss (wear) in terms of grams with respect to applied load min and temperature at the end of experimentation. The minimum mass loss of 0.103 g of material found lost during sliding wear with 5 N at 100°C and gradual material loss for increase in applied load and temperature were noticed in the graph as shown in Figure 7. However, for a particular point of process condition the mass loss found maximum increase of 0.306 g at applied load and temperature of 15 N and 150°C.

Figure 6 Coefficient of friction recorded for the sliding wear of the 3D built nickel alloy pin material (see online version for colours)



(a) (b)

(c)

For the same mass loss measured for different working condition is used to calculate the wear rate and the wear resistance calculated using a standard mathematical relation given in equations (2) and (3). The wear rate and wear resistance are inverse in calculations. Figure 8 shows the results of wear rate and wear resistance calculated for the proposed experimental design. It is clear to infer that the experimentation has wide variation with respect to the working conditions. In the proposed experimental design, the maximum wear resistance of 2,891.4 Nm/mm^3 is achieved at 100°C for 10 N applied load. In the same state of experimentation, the minimum resistance is noticed as 1,554.21 Nm/mm^3 at 200°C for 5 N applied load. The material at increase in temperature has yielded to maximum wear due to softening nature. The material behaviour the worn surface is subjected to surface characterisation studies using electron microscopy and spectroscopy.

Table 2 Mechanical properties of 3D printed nickel iron base superalloy developed through laser powder bed process

Properties	*Range measured*
Hardness	265.5 Hv
Density	8.2 g/cc
Tensile strength	1,195 MPa
Elongation	21.02%

Figure 7 Mass loss measured with respect to applied load and working temperature

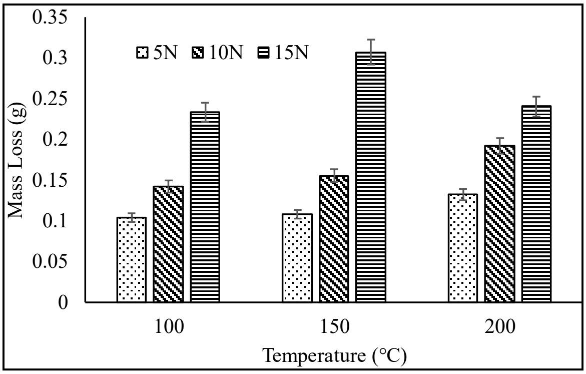
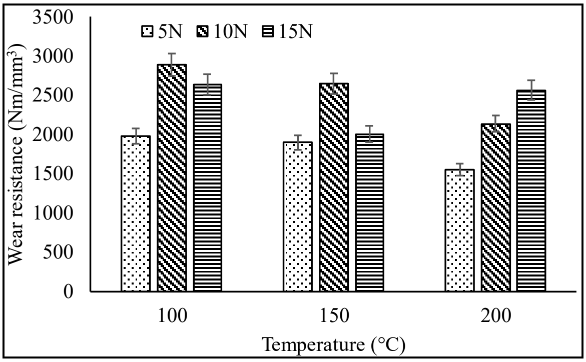


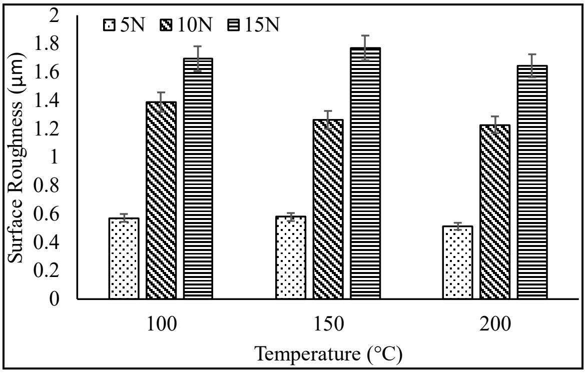
Figure 8 Wear rate and wear resistance mathematically calculated with respect to applied load and working temperature



3.2 *Surface analysis*

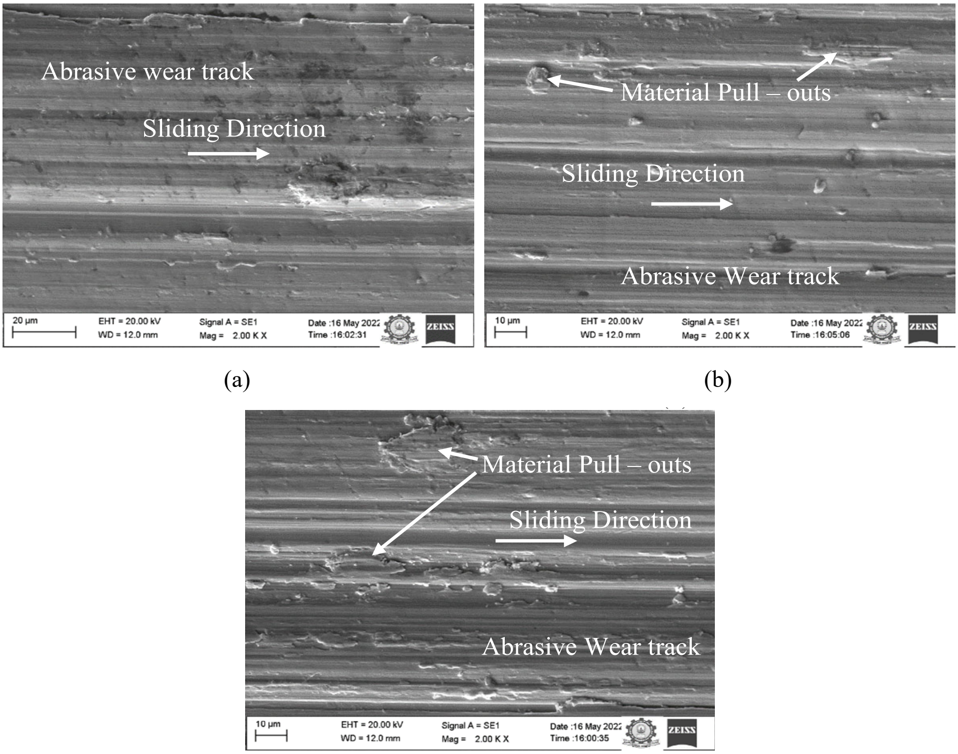
The behaviour of the 3D printed material after sliding wear experimentation are studied with respect to surface roughness on pin and electron microscopy for frictional wear analysis. The worn surface of the 3D printed pin is used to read the roughness with contact probe roughness metre. The surface roughnesses measured from the worn surface are as given in the graph (Figure 9) for comparison. While comparing the surface roughness and mass loss the material behaviour found similar in results derivation. The surface roughness of the worn pin found varying from 0.509 μm to 1.76 μm. Physically it is understood that surface of the pin and material loss are directly proportional. At minimum load of 5 N the worn surface roughness is in the range of 0.509 μm to 0.57 μm. With increase in mass loss has been influenced to produce catastrophic failure with high coefficient of friction as reported in Figure 6. Significance in roughness is caused due to high frictional force leading to surface damage. The worn surface is smooth and flat with less wear tracks for minimum applied load of 5 N at 200°C and slightly disturbed peaks for 200°C. Subsequently for the increase in load the worn surface revealed with wide variations in peaks and valley range. The distraction in peaks may be influenced due to the thermal effect and material susceptibility on sliding friction. In addition to study the material behaviour under the applied load and operating temperature, the worn surface are subject to metallurgical characterisation techniques.

Figure 9 Surface roughness measured on worn pin with respect to process condition



The surface topography of the tested 3D printed material, a scanning electron microscopy is used to reveal the worn surface. Figure 10 represents the sliding behaviour of the 3D printed material at different applied load conditions. At minimum load, the surface found with minimal wear tracks and the progression increased with increase in applied load. On continuous sliding between the pin and the disc for the predefined test condition, the wear found progressively increasing in terms of mass loss (Figure 7) as a result the surface degradation appeals. In depth of discussion the worn surface had severity in material loss due to abrasion and ablates of material in bulk. It is in the form of micro pull-out of materials increased due to applied load. In addition to the mechanical load and frictional (heat) energy, the effect of thermal load (100°C, 150°C and 200°C) has also influenced in surface quality of the material. Figures 11(a)–11(c) shows the EDS spectra for the worn surface at three different temperatures. At high temperature materials are prone to fail along with mechanical load and thermal effect in the form oxides. The spectra show the surface degradation of the material in the form of oxides. The along with the major elements the presence of oxygen on the surface found increasing. This is the hybrid action of mechanical and metallurgical transformation held over the contact surface. Especially the hard carbide may have some thermal effect and due to anisotropic in nature, the material starts to fail. In order to control material behaviour the post processing of 3D printed materials can be one the scope in future to lead the proposed work study.

Figure 10 Electron microscopic image of the pin-wear observed at different applied load (5 N, 10 N and 15 N) at a temperature (150°C), (a) 150°C – 5 N (b) 150°C – 10 N (c) 150°C – 15 N



(a) (b)

(c)

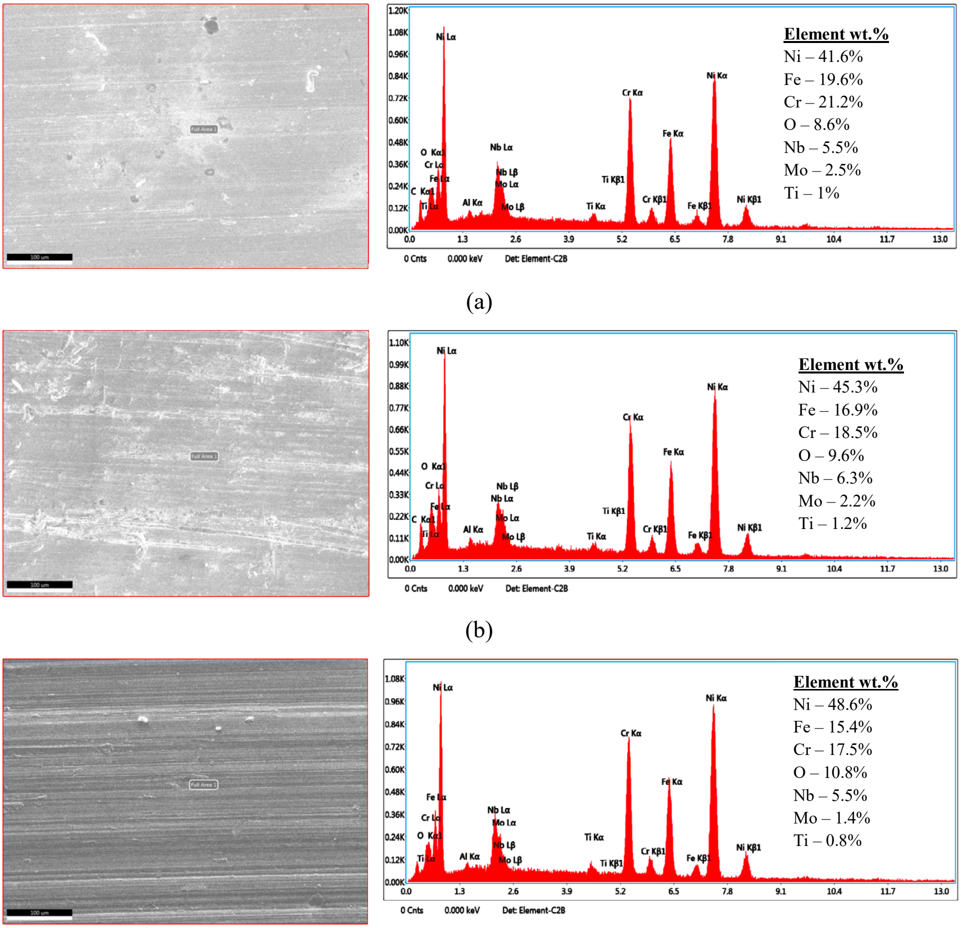
4 Discussion

From the experimental results, discussion and significant justifications are made to valid the data extracted. During the high temperature sliding wear, the material loss in terms of wear found increased with increase in temperature and applied load. The two major points identified from the wear analysis is

1 the initial damage was rapid and catastrophic to fracture the contact surface of 3D printed material

2 load applied and the temperature has impact on materials phase transformation towards a lead for material damage.

Figure 11 (a) Spectra analysis for the pin tested at 100°C with 10 N load (b) Spectra analysis for the pin tested at 150°C with 10 N load (c) Spectra analysis for the pin tested at 200°C with 10 N load (see online version for colours)



(a)

(b)

(c)

As a result, the materials under elevated temperature found sensitised and the material loss has highly influenced to drag the pin one disc. As the contact surface area between the pin and disc are mechanically distracted, the rate of friction will be increased simultaneously. It generally happens when there is an abrasion in pin or either adhesion in the disc sliding track. The sliding contact surface has catastrophic changes at increase in temperature borne to maximum wear due to softening nature. The material behaviour the worn surface reported with maximum surface roughness and surface damage with different forms of wear mechanism. Significant reason for the pull-outs is the 3D printed materials are non-homogeneous material and they possess high hardness due to localised heat affect. While the mechanical load is applied the bearing tendency of the structure found deformed and material failure occurred on the worn surface (Raj et al., 2022). As a result of these mechanical loading the surface of the material is susceptible at some occurrence and surface damage occurs due to frictional energy generated during the investigation.

5 Conclusions

The research on the 3D printed nickel base alloy has been evaluated in terms of mechanical properties and tribological properties. The observations and the results are keenly evaluated to find the recommendation with following points:

1 The density is 8.2 g/cc and hardness is 265.5 Hv which is very close to the conventional alloy (8.17 g/cc and 278 Hv). The mechanical strength 1,195 MPa which is near to the 1,240 MPa in commercial alloy. The difficulties in solidification has completed eradicate in 3D printing process and the similar properties are achieved.

2 The high temperature sliding wear analysis, the material loss, coefficient of friction and surface topography are studied in detail to recommend the material. It is clear that the material has minimum material loss of 0.103 g for minimal load of 5 N and maximum of 0.306 g which is negotiable at 15 N for 150°C. The material loss might be due to hybrid action of mechanical load and thermal effect generated in the defined working environment.

3 The surface roughness found to a maximum of 1.76 μm for 15 N which has made the material sensible and susceptible. The SEM and spectra analysis evidently proves that the surface had deformed with sliding tracks in the form of abrasive wear and oxide formation. There are some metal pull-outs as the 3D printed materials are anisotropic in nature.

Therefore, it is clear to recommend that the proposed material design has reasonable material loss and it is controllable on post of the developed material.

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