Chapter 6: Conclusions

The investigation on synthesis and characterization of Ni_3Al intermetallic compound, Cu-*h*BN hybrid material, Ni_3Al-based self-lubricating composites containing different combinations of solid lubricants, i.e., (a) Ni_3Al-10wt.% Ag, Ni_3Al-10wt.% WS_2, Ni_3Al-5wt.% Ag-5wt.% WS_2 composites, (b) Ni_3Al-10wt.% Ag, Ni_3Al-10wt.% Cu-*h*BN, Ni_3Al-5wt.% Ag-5wt.% Cu-*h*BN composites, and (c) Ni_3Al-10wt.% WS_2, Ni_3Al-10wt.% Cu-*h*BN, Ni_3Al-5wt.% WS_2-5wt.% Cu-*h*BN, as well as their friction and wear behaviour at a constant applied load of 10 N, sliding speed of 0.2 m/s and sliding duration of 360 m at different temperatures (RT, 200 °C, 400 °C, 600 °C and 800 °C) has resulted in some salient conclusions, which are presented as separate sub-sections namely,

6.1 Characterization of Ni_3Al intermetallic compound and (Cu-*h*BN) hybrid

1. The Ni_3Al intermetallic compound was successfully synthesized by ball milling. XRD analysis indicated the presence of the major peaks corresponding to the Ni_3Al intermetallic compound only.
2. The copper-modified or doped *h*BN nanosheet (Cu-*h*BN) hybrid nanomaterial could be successfully synthesized using ultrasound-assisted exfoliation and chemical processing of Cu salt with a reducing agent. Transmission electron microscopy confirmed the doping of CuO on *h*BN nanosheets, whereas XRD and XPS analyses established the presence of CuO, Cu_2O and *h*BN.

6.2 Characterization of Ni_3Al-based self-lubricating composites

3. The Ni_3Al-based self-lubricating composites, namely, Ni_3Al-10Ag, Ni_3Al-10WS_2, Ni_3Al 10Cu-*h*BN, Ni_3Al-5Ag-5WS_2, Ni_3Al-5Ag-5Cu-*h*BN and Ni_3Al-5WS_2-5Cu-*h*BN were successfully fabricated by vacuum hot press sintering technique. All the composite exhibited a fairly compact and dense microstructure with a uniform distribution of all its constituent elements.

4. XRD analysis of Ni_3Al-based self-lubricating composites revealed the presence of intense peaks corresponding to Ni_3Al apart from the peaks of Ag, WS_2, *h*BN and CuO, which confirmed that the powders are sintered under a vacuum environment without any oxidation or disintegration.
5. The addition of solid lubricants (Ag, WS_2) resulted in a decrease in the density of Ni_3Al composites. The incorporation of Ag to Ni_3Al decreased the hardness of Ni_3Al from 355 ± 5 HV to 313 ± 6 HV, whereas the addition of WS_2 to Ni_3Al raised the same from 355 ± 5 HV to 365 ± 5 HV in Ni_3Al-10WS_2. However, the addition of both silver and tungsten disulphide in Ni_3Al resulted in a hardness of 345 ± 8 HV. A decrease in the hardness of Ni_3Al-based composite with the addition of silver has been ascribed to the lower hardness and inherent soft nature of Ag. However, an increase in the hardness of composite containing WS_2 is due to its high hardness.
6. The density of Ni_3Al decreased with the incorporation of solid lubricants (Ag and Cu-*h*BN), either singly or in conjunction. The addition of Cu-*h*BN and Ag-Cu-*h*BN to the Ni_3Al intermetallic compound resulted in a decrease in its hardness from 355 ± 5 HV to 335 ± 5 HV and 325 ± 7 HV primarily because of the inherent softness of Ag and Cu-*h*BN indicating that the incorporation of soft phases reduces the brittleness of Ni_3Al and improves the ductility**.** However, the reduction in hardness is relatively more for the composite containing only Ag, i.e., Ni_3Al-Ag, than Ni_3Al-Cu-*h*BN or Ni_3Al-Ag-(Cu-*h*BN).
7. The addition of Cu-*h*BN resulted in a decrease in the hardness of Ni_3Al from 355 ± 5 HV to 335 ± 5 HV, whereas the addition of WS_2 and WS_2-Cu-*h*BN raised the hardness of Ni_3Al to 365 ± 6 HV and 358 ± 4 HV, which has been attributed to the respective hardness of *h*BN and WS_2.

6.3 Friction and wear behaviour of Ni_3Al-based self-lubricating composites

6.3.1 Tribological behaviour of $Ni_3Al/Ni_3Al\text{-}Ag/Ni_3Al\text{-}WS_2/Ni_3Al\text{-}Ag\text{-}WS_2$ composites

8. All the materials, i.e., Ni_3Al, Ni_3Al-10Ag, Ni_3Al-10WS_2 and Ni_3Al-5Ag-5WS_2, have shown a fluctuating trend of the variation of coefficient of friction (CoF) with time, have fluctuations in amplitude, which has been attributed to the initial roughness of the surfaces. The relatively larger fluctuations shown by Ni_3Al-10Ag in comparison to Ni_3Al, Ni_3Al-10WS_2 and Ni_3Al-5Ag-5WS_2 have been attributed to its relatively lower hardness, which allows the penetration of harder asperities of the hard Si_3N_4 ball causing abrasion.
9. All the composite materials, i.e., Ni_3Al, Ni_3Al-10Ag, Ni_3Al-10WS_2 and Ni_3Al-5Ag-5WS_2, have shown a decreasing trend in average CoF with increasing temperature. The CoF for Ni_3Al and Ni_3Al-10Ag has been found to decrease from 0.655 to 0.426 and from 0.453 to 0.259, respectively, whereas the CoF for Ni_3Al-10WS_2 and Ni_3Al-5Ag-5WS_2 has been observed to diminish from 0.421 to 0.239 and from 0.389 to 0.206 as the temperature is raised from RT to 800 °C. A decrease in CoF with increasing temperature has been attributed to the presence of a transfer layer containing solid lubricants Ag and WS_2 at relatively low temperatures and the existence of lubricious oxides (NiO, MoO_3, WO_3, Ag_2MoO_4, $Ag_2Mo_2O_7$ and $NiMoO_4$) at elevated temperatures on the worn surface of the composites as well as counterface ball.
10. The wear rate of Ni_3Al, Ni_3Al-10Ag, and Ni_3Al-10WS_2 has been observed to increase from 3.52 to 5.95 × 10^{-5} mm^3/Nm, 2.96 to 6.66 × 10^{-5} mm^3/Nm, and 1.582 to 2.52 x 10^{-5} mm^3/Nm, respectively, as the temperature is raised from RT to 400°C followed by a decrease to 2.46 × 10^{-5} mm^3/Nm, 2.3 × 10^{-5} mm^3/Nm and 1.06 × 10^{-5}

mm^3/Nm at 800°C. However, the wear rate of Ni_3Al-5Ag-5WS_2 composite has been observed to decrease continuously from 1.53 × 10^{-5} *mm^3/Nm* to 0.778 × 10^{-5} *mm^3/Nm* with increasing temperature from RT to 800 °C.

11. Among Ni_3Al, Ni_3Al-10Ag, Ni_3Al-10WS_2 and Ni_3Al-5Ag-5WS_2, the Ni_3Al-5Ag-5WS_2 composite has shown the lowest coefficient of friction (0.39-0.207) and wear rate (1.53-0.778 × 10^{-5} *mm^3/Nm*) among all the composites at all test temperatures (RT to 800 °C) due to synergetic effect of Ag and WS_2 over a wide temperature range. The improved tribological performance has been attributed to the formation and presence of NiO, MoO_3, $NiMoO_4$, silver molybdates, and WO_3 over the worn surface of composites and the counterface ball. At low temperatures, Ag and WS_2 operate as effective lubricants, whereas the formation of NiO, WO_3, $NiMoO_4$, Ag_2MoO_4, and $Ag_2Mo_2O_7$ at high temperatures by tribo-chemical reactions contribute to lowering the coefficient of friction as well as wear rate.
12. The wear mechanism for base Ni_3Al is abrasive and adhesive at RT and 200 °C, which changes to adhesion and delamination at 400 °C, and adhesion and oxidation at 600 °C and 800 °C. The wear mechanism for Ni_3Al-10Ag composite is abrasive and ploughing at RT, and abrasive and adhesive at 200 °C, which changes to adhesion and ploughing at 400 °C, and adhesion and oxidation at 600 °C and 800 °C. The wear mechanism for Ni_3Al-10WS_2 composite is abrasive at RT and 200 °C, and a mix of adhesion and delamination at 400 °C, whereas it is a mix of adhesion and oxidation at 600 °C and 800 °C. The Ni_3Al-5Ag-5WS_2 composite reveals abrasive and adhesive wear at RT and 200 °C, adhesion and ploughing at 400 °C, oxidation and delamination at 600 °C, and oxidation with glaze formation at 800 °C.

13. The occurrence of a synergistic action between Ag and WS_2 in providing effective lubrication over a broad range of temperatures offers an opportunity in preparing the Ni_3Al-based self-lubricating composites without comprising the hardness.

6.3.2 Tribological behaviour of Ni_3Al/Ni_3Al-Ag/Ni_3Al-Cu-*h*BN/Ni_3Al-Ag-Cu-*h*BN composites

14. Ni_3Al, Ni_3Al-10Ag, Ni_3Al-10Cu-*h*BN, and Ni_3Al-5Ag-5Cu-*h*BN composites have shown a fluctuating trend of the variation of coefficient of friction (CoF) with time. These fluctuations have been attributed to the initial surface roughness of the mating bodies. The relatively larger fluctuations shown by Ni_3Al-10Ag and Ni_3Al-5Ag-5Cu-*h*BN in comparison to Ni_3Al and Ni_3Al-10Cu-*h*BN have been attributed to its relatively lower hardness which allows the penetration of harder asperities of the hard Si_3N_4 ball causing abrasion.
15. The average CoF for Ni_3Al, Ni_3Al-10Ag, Ni_3Al-10Cu-*h*BN, and Ni_3Al-5Ag-5Cu-*h*BN has been found to decrease with increasing temperature. The CoF for Ni_3Al and Ni_3Al-10Ag decreased from 0.65 to 0.42 and from 0.45 to 0.26, respectively, whereas the CoF for Ni_3Al-10Cu-*h*BN and Ni_3Al-5Ag-5Cu-*h*BN reduced from 0.48 to 0.28 and from 0.41 to 0.19 as the temperature is raised from RT to 800 °C. A decrease in CoF with increasing temperature has been ascribed to the presence of an Ag-enriched transfer layer at low temperatures (RT to 400 °C) and lubricious *h*BN, Ag_2MoO_4, $Ag_2Mo_2O_7$, NiO, CuO, MoO_3, and $NiMoO_4$ enriched transferred layer at higher temperatures (600 °C and 800 °C) on both the worn surface of the composites and the counterpart Si_3N_4 ball.
16. The wear rate of Ni_3Al as well as the composites, has been found to increase as the temperature is raised from RT to 400 °C, followed by a decrease till 800 °C. The wear rate of Ni_3Al, Ni_3Al-10Ag, Ni_3Al-10Cu-*h*BN and Ni_3Al-5Ag-5Cu-*h*BN has

been observed to increase from 3.52 to 5.95 × 10^{-5} *mm^3/Nm*, 2.96 to 6.66 × 10^{-5} *mm^3/Nm*, 2.285 to 3.1 × 10^{-5} *mm^3/Nm*, and 2.185 to 2.95 × 10^{-5} *mm^3/Nm*, respectively, as the temperature is raised from RT to 400 °C followed by a decrease to 2.46 × 10^{-5} *mm^3/Nm*, 2.3 × 10^{-5} *mm^3/Nm*, 1.95 × 10^{-5} *mm^3/Nm* and 1.85 × 10^{-5} *mm^3/Nm* at 800°C.

17. Among Ni_3Al, Ni_3Al-10Ag, Ni_3Al-10Cu-*h*BN and Ni_3Al-5Ag-5Cu-*h*BN, The Ni_3Al-5Ag-5Cu-*h*BN composite exhibited the lowest CoF (0.41 to 0.19) and wear rate (2.185 to 1.85 × 10^{-5} *mm^3/Nm*) at all the temperatures and this has been ascribed to the synergistic lubrication effect between Ag and Cu-*h*BN from RT to 800 °C. The improved tribological performance has been attributed to the presence of lubricious species (Ag, *h*BN, Ag_2MoO_4, $Ag_2Mo_2O_7$, NiO, CuO, MoO_3, and $NiMoO_4$) over the worn surface of composites and the counterface ball.
18. The wear mechanism for Ni_3Al-10Cu-*h*BN composite is abrasive and delamination at RT and 200 °C, which changes to a mix of adhesion and delamination at 400 °C, oxidation and adhesion dominated wear at 600 °C, and tribo-oxidation at 800 °C. For Ni_3Al-5Ag-5Cu-*h*BN composite, the wear mechanism is abrasive, adhesive and ploughing at RT and 200 °C, adhesion and ploughing at 400 °C, adhesion and tribo-oxidation at 600 °C, and oxidation with glaze formation at 800 °C.
19. The synergistic action between Ag & Cu-*h*BN and the improved bonding characteristics of Cu-*h*BN with the matrix of Ni_3Al are believed to have played a vital role in extending the self-lubrication capability of Ni_3Al-10Cu-*h*BN and Ni_3Al-5Ag-5Cu-*h*BN composites without compromising the hardness.

6.3.3 Tribological behaviour of Ni_3Al/Ni_3Al-WS_2/Ni_3Al-Cu-*h*BN/Ni_3Al-WS_2-Cu-*h*BN composites

20. All the materials, i.e., Ni_3Al, Ni_3Al-10WS_2, Ni_3Al-10Cu-*h*BN and Ni_3Al-5WS_2-5Cu-*h*BN composites, have shown a continuous decrease in CoF with increasing temperature. The CoF for Ni_3Al and Ni_3Al-10WS_2 has been found to decrease from 0.65 to 0.42 and 0.42 to 0.24, respectively, with increasing temperature from RT to 800°C. Similarly, the CoF for Ni_3Al-10Cu-*h*BN and Ni_3Al-5WS_2-5Cu-*h*BN has been found to reduce from 0.48 to 0.28 and 0.44 to 0.18, respectively. A decrease in CoF with increasing temperature has been attributed to the presence of a transfer layer containing solid lubricants WS_2 at relatively low temperatures and the existence of *h*BN and lubricious oxides (NiO, MoO_3, WO_3, CuO and $NiMoO_4$) at elevated temperatures on the worn surface of the composites as well as counterface ball.
21. The wear rates of Ni_3Al, Ni_3Al-10WS_2, Ni_3Al-10Cu-*h*BN and Ni_3Al-5WS_2-5Cu-*h*BN composites have been observed to increase from 3.52 to 5.95 × 10^{-5} mm^3/Nm, 1.58 to 2.52 × 10^{-5} mm^3/Nm, 2.285 to 3.1 × 10^{-5} mm^3/Nm, and 2.12 to 2.61 × 10^{-5} mm^3/Nm, respectively, as the temperature is increased from RT to 400 °C followed by a decrease to 2.46 × 10^{-5} mm^3/Nm, 1.06 × 10^{-5} mm^3/Nm, 1.95 × 10^{-5} mm^3/Nm, and 0.875 × 10^{-5} mm^3/Nm, respectively, at 800 °C.
22. Ni_3Al-5WS_2-5Cu-*h*BN has shown the lowest (μ ~ 0.44 to 0.18, W ~ 2.61 × 10^{-5} to 0.875 × 10^{-5} mm^3/Nm) at all the temperatures except at RT, 200 °C, and 400 °C, at which Ni_3Al-10WS_2 has shown a slightly lower CoF and wear rate in comparison to Ni_3Al-5WS_2-5Cu-*h*BN. The improved tribological performance of Ni_3Al-5WS_2-5Cu-*h*BN reflects the cooperative lubricating action among CuO, *h*BN, NiO, MoO_3, WO_3 and $NiMoO_4$ in reducing friction and wear at temperatures beyond 400 °C.

However, no assistive action could be observed between WS_2 and Cu-*h*BN at temperatures below 400 °C.

23. The wear mechanism for Ni_3Al-5WS_2-5Cu-*h*BN composite is abrasive and delamination at RT and 200 °C, a mix of ploughing, delamination and adhesion at 400 °C, adhesion and tribo-oxidation at 600 °C, and formation of a smooth and continuous glaze formation at 800 °C.
24. Copper-modified *h*BN nanosheet (Cu-*h*BN) hybrid material offers an alternative to be used as a solid lubricant either singly or in conjunction with other solid lubricants for various applications involving relative sliding motion over a range of temperatures.
25. A synergistic action between WS_2 and Cu-*h*BN in providing effective lubrication over a broad range of temperatures offers an opportunity to prepare the Ni_3Al-based self-lubricating composites without compromising the hardness.

6.4 Comparative analysis of tribological behaviour of Ni_3Al-Ag-WS_2, Ni_3Al-Ag-Cu-*h*BN and Ni_3Al-WS_2-Cu-*h*BN composites

26. All the composites containing a combination of solid lubricants, i.e., Ni_3Al-Ag-WS_2, Ni_3Al-Ag-Cu-*h*BN and Ni_3Al-WS_2-Cu-*h*BN, have been observed to exhibit the occurrence of a synergistic action between the constituent solid lubricants which helps in lowering the coefficient of friction and wear at elevated temperatures in comparison to the composites containing single lubricant (Ag, WS_2 and Cu-*h*BN) and Ni_3Al. The tribological performance of these composites in terms of the average coefficient of friction and the wear rate fall in the same band. However, Ni_3Al-Ag-WS_2 composite performed marginally better than Ni_3Al-WS_2-Cu-*h*BN and Ni_3Al-Ag-Cu-*h*BN composites.

Future scope

In the present study, the potential of a combination of solid lubricants viz Ag-WS_2, Ag-(Cu-*h*BN), WS_2-(Cu-*h*BN) has been evaluated in extending the regime of effective lubrication of Ni_3Al-based self-lubricating composites at a constant load and sliding speed of 10 N and 0.2 m/s at different temperatures (RT, 200 °C, 400 °C, 600 °C and 800 °C under non-conformal (ball-on-disc) contact conditions. However, the components may have to work under different conditions of load and speed as well in the applications where the components have to work under conformal contacts. Hence, future studies may be conducted to explore the frictional performance at different loads and speeds under conforming conditions to explore their tribological behaviour. Some of the below-mentioned studies may also be undertaken in future.

1. The tribological performance of the composites may be assessed under different loads and speeds to have a better understanding on their friction and wear behaviour over a wide range of applications.
2. The friction and wear characteristics of composites may be explored under conformal contact conditions with or without texturing to analyze the effect of texturing on the tribological performance of composites.
3. The composites could be fabricated using alternative fabrication methods such as Spark Plasma Sintering (SPS) and Microwave sintering. Their tribological performance may be evaluated and compared under same conditions of load, speed, and temperature.
4. Different combinations of solid lubricants (Ag, MoS_2/WS_2, Graphene, Ni-coated *h*BN and Max phase-Ti_3SiC_2) may be used to prepare Ni_3Al composites and their friction and wear characteristics may be examined at RT, 200 °C, 400 °C, 600 °C, 800 °C and 1000 °C.