Chapter 8: CONCLUSIONS

The present investigation on synthesis and tribological characterization of Ni alloy-based self-lubricating composites containing different combinations of solid lubricants, i.e., (a) Ni alloy-10 wt.% Ag (NA), Ni alloy -10 wt.% Ag -1wt.% rGO (NAG), Ni alloy -10 wt.% Ag -1wt.% rGO-Ni (NANG) composites, (b) Ni alloy-10 wt.% Ag-(0.5, 1.0, 1.5, and 2.0 wt.%) rGO-Ni composites namely, NANG0.5, NANG1.0, NANG1.5, and NANG2.0 (c) Ni alloy-10 wt.% Ag-(2, 4, 6, and 8 wt.%) *h*-BN-Ni composites, namely, NAh2, NAh4, NAh6, and NAh8, and (d) Ni alloy-10 wt.% Ag- 1 wt.% rGO-Ni- (2, 4, 6, and 8 wt.%) *h*-BN-Ni composites, namely, NANGh2, NANGh4, NANGh6, and NANGh8 at a constant applied load of 5 N, sliding speed of 0.5 m/s and sliding duration of 1200 m at different temperatures (RT, 200, 400, 600, and 800 °C) has resulted in the following salient conclusions, which are presented as separate sub-sections depending on the combination of solid lubricants.

8.1 Ni ALLOY-BASED COMPOSITES CONTAINING Ag, Ag-rGO, AND Ag-(rGO-Ni)

1. Both rGO and Ni-doped rGO could be successfully synthesized via wet chemistry route. TEM, XRD, Raman spectroscopy, and XPS analyses confirmed the doping of Ni nanoparticles in rGO lamellae.
2. Nickel base alloy (N0) and composites could be successfully fabricated by spark plasma sintering technique. The doping of rGO by Ni resulted in a nearly uniform distribution in the matrix in comparison to undoped rGO, which showed some agglomeration.
3. The hardness of the Ni base alloy decreased from 515 ± 5 to 496 ± 4 and 502 ± 6 $HV_{0.3}$ by incorporating solid lubricant(s) such as Ag and Ag-rGO. However, a combination of Ag-(rGO-Ni) raised the hardness as to 510 ± 3 $HV_{0.3}$, i.e.,

approximately to the same level of the base alloy due to the improved wetting characteristics of caused by the doping of rGO by Ni.

4. The average CoF for N0 and NA has been found to decreased with increasing temperature from RT to 800 ^{o}C. The CoF for NAG and NANG decreased from RT to 600 ^{o}C followed by a slight increase at 800 ^{o}C. The change in CoF with increasing temperature has been attributed to the presence of a transfer layer containing solid lubricants Ag at relatively low temperatures and the existence of lubricious oxides (NiO, MoO_3, Ag_2MoO_4, $Ag_2Mo_2O_7$ and $NiMoO_4$) at elevated temperatures on the worn surface of the composites.
5. The wear rate of N0 increased from RT to 600 ^{o}C followed by a decrease at 800 ^{o}C. However, the wear rate of NA increased with increasing temperature from RT to 400 ^{o}C and decreased thereafter till 800 ^{o}C. The wear rate of NAG increased from RT to 200 ^{o}C followed by a decrease till 800 ^{o}C and that of NANG increased from RT to 200 ^{o}C and decreased at 600 ^{o}C before increasing again to at 800 ^{o}C. The observed variation has been explained on the basis of morphology of the worn surfaces of the composites and presence of lubricious oxides (NiO, MoO_3, Ag_2MoO_4, $Ag_2Mo_2O_7$ and $NiMoO_4$).
6. NANG showed the lowest CoF and wear rate from RT to 600 ^{o}C which has been ascribed to the occurrence of a synergistic action between Ag and rGO-Ni in assisting each other’s lubrication capability apart from its relatively higher hardness as compared to other materials.
7. The combination of Ag-rGO or Ag-(rGO-Ni) failed to offer any assistive action in providing effective lubrication beyond 600 ^{o}C, probably due to the loss of lubricity of rGO and non-formation silver molybdate (Ag_2MoO_4).
8. The dominant wear mechanisms for N0 and NA have been found to be ploughing and delamination from RT to 400 ^{o}C, which changed to oxidative wear at 600 and 800 ^{o}C. However, the wear mechanisms for NAG and NANG were ploughing

and delamination from RT to 200 ^{o}C, delamination at 400 ^{o}C and a combination of abrasion and oxidation at 600 and 800 ^{o}C as revealed by the worn surface morphologies at these temperatures.

8.2 COMPOSITES CONTAINING FIXED AMOUNT OF Ag AND DIFFERENT AMOUNTS OF rGO-Ni

9. The Ni alloy-based self-lubricating composites, namely, NANG0.5, NANG1.0, NANG1.5, and NANG2.0 were successfully fabricated by spark plasma sintering technique. All the composites exhibited a fairly compact and dense microstructure with nearly uniform distribution of solid lubricants.
10. The hardness of the composites increased from 500 ± 2 to 517 ± 1 $HV_{0.3}$ with increasing amount of rGO from 0.5 to 2.0 wt.%, and a combination of Ag-2.0 wt.% rGO resulted in approximately the same hardness as that of the base alloy due to improved wettability of rGO caused by the doping of rGO by Nickel.
11. The average CoF for NANG0.5, NANG1.0 and NANG1.5 decreased significantly from RT to 200 ^{o}C before increasing marginally from 200 to 400 ^{o}C and decreased slightly from 400 to 600 ^{o}C before increasing again at 800 ^{o}C. However, the CoF for NANG2.0 remained almost stable from 200 to 800 ^{o}C after a decrease from RT to 200 ^{o}C and it showed the lowest at all the temperatures except 800 ^{o}C.
12. The wear rate for NANG0.5, NANG1.0, and NANG1.5 increased marginally with increasing temperature from RT to 200 ^{o}C and then decreased till 600 ^{o}C before increasing again at 800 ^{o}C. However, the wear rate of NANG2.0 remained almost the same from RT to 200 ^{o}C with a slight decrease thereafter to 600 ^{o}C, which was followed by a sharp increase at 800 ^{o}C.
13. Both the average CoF and the wear rate of the composites decreased with

increasing amount of rGO-Ni from 0.5 to 2.0 wt.% at all the temperatures. NANG2.0 has shown the lowest CoF as well as wear rate from RT to 600 ^{o}C due to the occurrence of a synergistic action between Ag and rGO-Ni.

14. The increase in both the CoF and the wear rate after 600 ^{o}C for the composites containing Ag and rGO has been ascribed to the probable loss of assistive action between rGO and other lubricating silver molybdates or oxides.
15. Based on the observed friction and wear performance, it can be concluded that a Ni based alloy containing an optimised combination of Ag and rGO can be a potential tribo-material for use in temperatures ranging from RT to 600 ^{o}C.
16. The dominant wear mechanisms for NANG0.5, NANG1.0, and NANG1.5 have been found to be ploughing and delamination from RT to 200 ^{o}C, delamination at 400 ^{o}C, and a combination of abrasion and oxidation at 600 and 800 ^{o}C. However, ploughing and abrasion from RT to 600 ^{o}C and oxidation at 800 ^{o}C have been found to be the dominant mechanisms for NANG2.0.

8.3 COMPOSITES CONTAINING FIXED AMOUNT OF Ag AND DIFFERENT AMOUNTS OF *h*-BN-Ni

17. The hybrid material, i.e., Ni-doped *h*-BN (*h*-BN) has been successfully synthesized via wet chemistry route. TEM along with XRD and XPS analyses confirmed the doping of Ni nanoparticles on *h*-BN nanosheets.
18. A homogeneous distribution of *h*-BN was observed for 2 and 4 wt.% addition of *h*-BN in NA. However, an agglomeration of *h*-BN was seen in the composites containing 6 and 8 wt.% *h*-BN. The hardness of the composites decreased from 405 ± 3 to 313 ± 2 $HV_{0.3}$ with an increase in addition of *h*-BN from 2 to 8 wt.% due to either the soft nature of *h*-BN or its agglomeration.
19. The average CoF for all the composites i.e., NAh2, NAh4, NAh6 and NAh8

decreased with increasing temperature as well as the amount of addition of *h*-BN-Ni. This has been credited to the occurrence of a synergistic action between Ag and *h*-BN and the presence of lubricious compounds on the worn surface of composites.

20. The wear rate for the composites, i.e., NAh2, NAh4, NAh6, and NAh8 has been found to increase from RT to 400 ^{o}C, which is followed by a decrease at 600 and 800 ^{o}C.
21. At a particular temperature, the wear rate of the composites decreased with the addition of *h*-BN from 2 and 4 wt.%, whereas the addition of 6 and 8 wt.% resulted in an increase in the wear rate. The observed behaviour has been attributed to the characteristics of the transfer layer present on their worn surfaces apart from the synergistic action of Ag and *h*-BN and the relative dominance of reducing hardness and the lubricating efficacy of the combination of lubricants.
22. The dominant wear mechanisms for all the composites i.e., NAh2, NAh4, NAh6, and NAh8 have been found to be (i) ploughing at RT, (ii) a combination of abrasion and ploughing at 200 and 400 ^{o}C and (iii) oxidation at 600 and 800 ^{o}C.

8.4 COMPOSITES CONTAINING FIXED AMOUNTS OF Ag, rGO-Ni, AND DIFFERENT AMOUNTS OF *h*-BN-Ni

23. All the composites containing Ag, rGO-Ni and different amount of *h*-BN-Ni i.e., NANGh2, NANGh4, NANGh6, and NANGh8 could be successfully prepared by spark plasma sintering technique. All the composites exhibited a fairly compact and dense microstructure with a uniform distribution of lubricating agents.
24. The hardness of the composites has been found to decrease from 458 ± 3 to 412 ± 2 $HV_{0.3}$ with the increasing amount of *h*-BN from 2 to 8 wt.% which has been ascribed to the inherent softness of *h*-BN.

25. The CoF of composites decreased with increasing content of *h*-BN from 2 to 8 wt.% with a marginal change in wear rate at all the temperatures. However, composite containing 8 wt.% *h*-BN, showed the lowest CoF (~ 0.27- 0.17) and wear rate (~5.1 - 6.5 × 10^{-5} mm^3/Nm) at all the temperatures which has been credited to the relatively larger amount of *h*-BN and the synergistic action among Ag, rGO, and *h*-BN.
26. A novel combination of solid lubricants e.g., Ag, Ni-doped rGO, and Ni-doped *h*-BN in Ni alloy-based composites was able to provide low friction (~ 0.39-0.17) and low wear properties (~ 4.4-8.4 ×10^{-5} mm^3/Nm) under extended regime of temperatures from RT to 800 ^oC.
27. A combination of abrasion and ploughing at RT and 200 ^oC, delamination, ploughing, and oxidation at 400 ^oC whereas oxidation at 600 and 800 ^oC have been found to be the dominant mechanisms of wear.
28. On the basis of the present investigation, it can be concluded that among all the hybrid composites containing a combination of two or more solid lubricants, NANGh8 having 10 wt. %Ag-1 wt.% Ni doped rGO-8 wt. % Ni doped *h*-BN has shown the lowest coefficient of friction (~ 0.27- 0.17) from RT to 800 ^oC under the conditions used in the present study and it can be used as a potential candidate where achieving and maintaining a low friction is of major concern. However, if the material loss due to wear is a concern, NANG2.0 containing 10 wt. % Ag-2 wt.% Ni doped rGO can be a potential combination as it has exhibited the lowest wear rate from RT to 600 ^oC.