CHAPTER 6

CONCLUSIONS

Based on the experimental studies reported in the chapters of this thesis, the consolidated conclusions drawn are presented in this chapter, which is followed by the scope for future study.

6.1 SYNTHESIS AND TRIBOLOGICAL BEHAVIOUR OF MULTI-LAYER GRAPHENE COATINGS

6.1.1 SYNTHESIS OF MULTI-LAYER GRAPHENE COATINGS

1. Graphene films have been successfully synthesised directly on bearing steel catalysed with a nickel layer using thermal chemical vapour deposition at different growth temperatures following the mechanism of precipitation and growth. Raman spectra confirmed the deposition of multi-layer graphene films through the presence of three characteristics peaks, namely, D, G, and 2D peaks centred around ~ 1350 cm^{-1}, ~ 1580 cm^{-1}, and ~ 2700 cm^{-1}, respectively.

2. The I_D/I_G ratio has been observed to decrease with increasing growth temperature from 650 to 850 °C indicating an improvement in the quality of graphene with increasing temperature with no significant change in it beyond 850 °C. Hence, a temperature of 850 °C has been used as the optimum temperature for the growth of graphene on the basis of the lowest I_D/I_G and the highest I_{2D}/I_G observed for this temperature.

3. The suppression of 2D and G peak intensities and the strengthening of D peak have been found to occur with increasing acetylene flow rate from 6 to 10 sccm. The maximum I_{2D}/I_G and the minimum I_D/I_G ratios observed for 6 sccm suggested that the best quality graphene has been grown for 6 sccm at a growth temperature of 850 °C.

4. A relatively higher I_{2D}/I_G ratio for the graphene grown under a reaction time of 10 min in comparison to that for 20 min indicates that comparatively thinner graphene films have been synthesised at relatively shorter reaction times.

5. The thickness of graphene layers grown under optimised conditions, i.e., 850 °C growth temperature, 6 sccm acetylene flow rate and 10 min reaction time, is about 21 ± 2 nm.

6.1.2 TRIBOLOGICAL BEHAVIOUR OF MULTI-LAYER GRAPHENE COATINGS

6.1.2.1 Unidirectional Sliding

6. For the initial 800 cycles, the coefficient of friction is found to be ~ 0.89 for the steel-base steel tribo-pair, whereas ~ 0.66 for steel-nickel plated steel tribo-pair. However, a significant reduction in the coefficient of friction has been observed to occur for steel slid against multi-layer graphene-coated steel with an average value of ~ 0.15, which has been attributed to the lubricating effect of graphene.

7. The maximum wear rate and wear volume observed for steel ball slid against base steel, and the least values for the same observed for steel ball slid against graphene-coated steel, indicate the effectiveness of the multi-layer graphene coating in reducing the friction and wear of the system.

8. A significant reduction in the coefficient of friction and wear volume for steel-graphene coated steel tribo-pair has been attributed to the existence of graphene on steel surface even after sliding tests and the transfer of the graphene onto the counterface ball, which protects the underlying materials from direct metal-to-metal contact apart from providing a low shearing interface between the mating bodies.

9. For longer duration (5600 cycles), a significant reduction in the coefficient of friction has also been observed for the steel against multi-layer graphene-coated steel from the beginning of the test, and an average value of ~ 0.18 has been recorded of the coefficient of friction throughout the friction test, which is slightly more than that of short duration (800 cycles) sliding and the same has been attributed to the loss of the beneficial effect of graphene coatings with time.

10. A reduction of the wear volume and wear rate of 2 and 3 order respectively, observed for the ball slid against multi-layer graphene-coated steel, has been ascribed to the ability of graphene coatings to provide effective lubrication even for a longer duration under atmospheric conditions.

6.1.2.2 Reciprocating Sliding

11. For the initial 600 cycles, a significant reduction in the coefficient of friction has been found to occur for the steel ball against multi-layer graphene-coated steel, which has shown a coefficient of friction of ~ 0.15, that is approximately five times lower than that observed for steel-base steel tribo pair, i.e., ~ 0.74.

12. The average coefficient of friction has been found to increase with increasing load for steel ball against base steel as well as against nickel-plated steel. However, the average coefficient of friction of steel against multi-layer graphene-coated steel is found to decrease the increasing normal load from 0.1 to 0.5 N, followed by an increase for 1 N, which has been attributed to the failure of the graphene coating during the sliding test.

13. The minimum average coefficient of friction of ~ 0.13 is observed for 1 N load till 250 cycles for steel ball against multi-layer graphene-coated steel. However, it shoots up sharply due to the failure of the graphene coating after 250 cycles to a value of ~ 0.9 before attaining a value of ~ 0.6 for the remaining duration of the test.

14. The wear volume has been observed to increase with increasing load for the steel balls slid against base and nickel-plated steel due to enhanced possibility of direct metal-to-metal contact, whereas the wear volume of the ball slid against multi-layer graphene-coated steel is found to decrease with increasing load from 0.1 to 0.5 N followed by a slight increase for 1 N.

15. A decrease in the coefficient of friction and wear volume with increase in load from 0.1 to 0.5 N for steel slid against multi-layer graphene-coated steel has been attributed to the homogenisation and smoothening of graphene coatings with increasing load and the transfer of graphene to the ball. However, an increase in the coefficient of friction and wear volume for normal load of 1 N has been ascribed to the failure of graphene coating and initiation of direct metal-to-metal contact.

16. The existence of graphene on the steel disc surface and the transfer of graphene to the counter surface after the reciprocating dry sliding friction tests have been confirmed by Raman spectroscopy.

17. TEM images of the disc and ball surfaces (steel ball vs multi-layer graphene-coated steel) after the sliding friction tests have shown the presence of 10 ± 2 nm and 5 ± 2 nm thick layers of graphene respectively, along with the fragments of graphene sheets and amorphous carbon.

18. The wear mechanisms for steel-base steel is the combination of adhesion, abrasion with wear induced oxidation, whereas the wear mechanisms associated with steel-nickel plated steel tribo-pair is a mixture of adhesion and oxidation. However, the abrasion is the dominant mechanism of wear for steel-graphene coated steel tribo-pair for both unidirectional and reciprocating sliding.

6.2 TRIBOLOGICAL BEHAVIOUR OF GRAPHENE AS AN ADDITIVE IN WATER

19. Water-based lubricant containing different concentrations of (0.01, 0.05, 0.1, 0.5 wt. %) of graphene oxide has been successfully synthesised with the thickness of dispersed sheets about ~ 0.12 nm and the size less than 5 μm. The viscosity of water-graphene oxide dispersions has been found to increase with increasing amount of graphene oxide in water.

20. The tribo-pair slid under pure water has shown a higher coefficient of friction (~ 0.56) with relatively larger amplitude of fluctuations throughout the friction test. However, a significant reduction in the coefficient of friction has been found to occur by addition of just a small concentration of graphene oxide, which has been attributed to the adsorbtion of graphene nano-sheets and subsequent formation of a protective tribo-film on the sliding surfaces leads to a smoother contact between the mating bodies.

21. The average coefficient of friction and wear volume have been found to decrease with the increasing amount of graphene oxide from 0.01 to 0.1 wt. % followed by a slight increase for 0.5 wt. %, indicating that 0.1 wt. % is the optimum content of graphene oxide under the conditions used in the present investigation. The lowest coefficient of friction of ~ 0.12 has been observed for 0.1 wt. % of graphene oxide in water.

22. The reduction in coefficient of friction and wear volume for graphene oxide-water dispersion has been attributed to the formation of a tribo-layer probably of graphene oxide (GO) by adsorbtion of nano-sheets on the disc as well as ball surface, which hinders the direct metal-to-metal contact and provides low shearing properties at the interface.

23. The lower and higher concentrations of graphene oxide in water have shown relatively higher coefficients of friction and wear volume in comparison to 0.1 wt. %. For the concentration lower than 0.1 wt. %, the graphene oxide sheets in water are not sufficient enough to make a continuous layer over the friction surface, whereas agglomeration of graphene oxide nano-sheets occurs for the highest concentration of 0.5 wt. %, and hinders the formation of a well-spread tribo-layer on the sliding interface.

24. The addition of graphene oxide in water results in improved tribological performance in terms of both the reduced coefficient of friction and wear volume at relatively lower loads compared to that at higher loads due to the loss of effectiveness of tribo-layer in preventing the asperity-asperity interactions under high contact pressures.

25. Both the coefficient of friction and wear volume have been found to decrease with increasing sliding speed up to 0.05 m/s followed by a slight increase for 0.1 m/s lubricated with 0.1 wt. % graphene oxide in water. The sliding speed of 0.05 m/s has shown the least coefficient of friction and wear volume due to better circulation of dispersed nano-sheets in water and thus, enhancing the formation of tribo-layer.

To summarise, the multi-layer graphene coating has excellent potential for improving the friction and wear behaviour of steel-steel tribo-pairs under atmospheric condition. The results also demonstrate that graphene oxide as an additive in water can be a promising choice in improving the tribological performance of water as an environmental friendly lubricant and the potential needs to be tapped for a cleaner, greener and sustainable environment.