Chapter 2: REVIEW OF LITERATURE

This chapter starts with a summary of composites, their types and fabrication approaches with the emphasis on spark plasma sintering (SPS). Additionally, the chapter offers a succinct review of the concept of wear, its different types, and the variables affecting wear and lubrication (liquid and solid). The need of solid lubrication is discussed along with a brief description of different types of solid lubricants. This is followed by an exhaustive review of literature on the tribological behaviour of Ni alloy-based high temperature self-lubricating composites containing different solid lubricants. The research gap is identified on the basis of the critical assessment of the literature and the problem statement, and the research objectives are presented at the end of the chapter.

2.1 COMPOSITE AND ITS TYPES

Composite material is a macro-physical integration of two or more, physically and or chemically, different phases in order to achieve superior characteristics such as high stiffness, high strength, high Young's modulus, high corrosion resistance, low density, high thermal/ electrical conductivity and high wear resistance compared to constituent materials. It consists of two phases: matrix (continuous) and reinforcement (discontinuous and present in smaller quantities). The matrix is a relatively soft phase having specific physical and mechanical properties such as ductility, formability, and thermal conductivity, whereas reinforcement is a hard phase having high strength, high stiffness, and low thermal expansions [2]. They can be categorized in three ways: (i) matrix (metal/polymer/ceramic), (ii) phase of reinforcement (long or short fiber/ whiskers/ particulate), and (iii) geometry of reinforcement (continuous fiber/ discontinuous fiber/ wire) as depicted in Fig. 2.1. The main focus of the present investigation is on the MMCs (metal matrix composites) due to their lower density, increased specific strength, and stiffness, increased high temperature performance limits, and improved wear-abrasion resistance compared to CMCs (ceramic matrix composites) and PMCs (Polymer matrix composites). MMCs consist of a continuous metallic matrix (i.e., Al, Cu, Mg, Ni, Mg, and Ti etc) and a reinforcement phase, which can be either dispersed ceramics (i.e., oxides, carbides) or metallic phases (i.e., Ag, W, Mo, and Pb etc.) consisting of ~ 50% of the total volume of the matrix material.

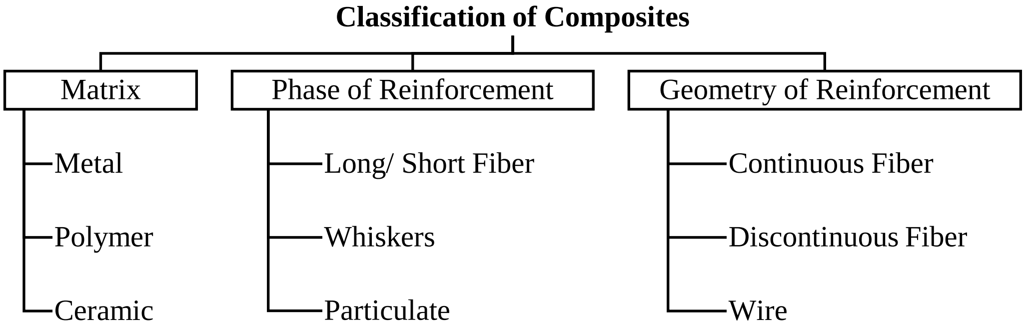

Fig. 2. 1 Classification of Composites

The composites need to be incorporated with at least 10% reinforcement to enhance their properties significantly. Particulate-reinforced composites include particles like spheres, rods, and flakes, with whisker reinforcements also grouped under this category. These are "discontinuous" reinforcements due to their lower volume fractions. Continuous fiber-reinforced composites have fibers much longer than their cross-sections. If composite properties vary with fiber length, they are called discontinuous fiber composites; if not, they are continuous fiber-reinforced. Continuous fiber composites have fibers as long as the composite part, with each layer oriented for specific properties. Laminated composites achieve isotropy in a plane through controlled fiber orientation and may include hybrid laminates like glass/epoxy with aluminium for enhanced resistance properties. Weaving, braiding, or knitting fiber bundles create interlocking fibers with properties in the third dimension, easier handling, lower cost, and better conformity to shapes.

MMCs are extensively employed in a variety of applications since they exhibit remarkable characteristics such as high toughness, high specific modulus, high thermal conductivity, and reduced thermal coefficient of expansion, among others. They possess higher temperature tolerance, higher transverse strength, higher shear strength, better compressive strength, and superior fire resistance when compared to PMCs. Better toughness, moisture resistance, high electrical and thermal conductivity, resilience to thermal shock, and ease of joining, shaping, and manufacturing are just a few of the benefits that MMCs have over CMCs.

2.2 PROCESSING TECHNIQUES FOR COMPOSITES

The routes for fabrication of MMCs can be classified into four primary processes on the basis of physical state of the matrix material during the processing stage as depicted in Fig. 2.2.

2.2.1 Liquid State Processing

The fabrication of the MMCs using liquid state processing involves mixing and casting molten metal with a reinforcing phase. This method is divided into three categories: **melt stirring, gas pressure infiltration, and squeeze casting**. **(i) Melt stirring,** the reinforcing phase is mixed into the molten metal with a stirrer before solidifying. This method is simple but can degrade the reinforcement at high temperatures. **(ii) Gas pressure infiltration** involves the injection of molten metal under high gas pressure into a ceramic preform and production of pore-free castings. **(iii) Squeeze casting**, molten metal is pressed into a preform held at the bottom of the setup by applying pressure using ram displacement.

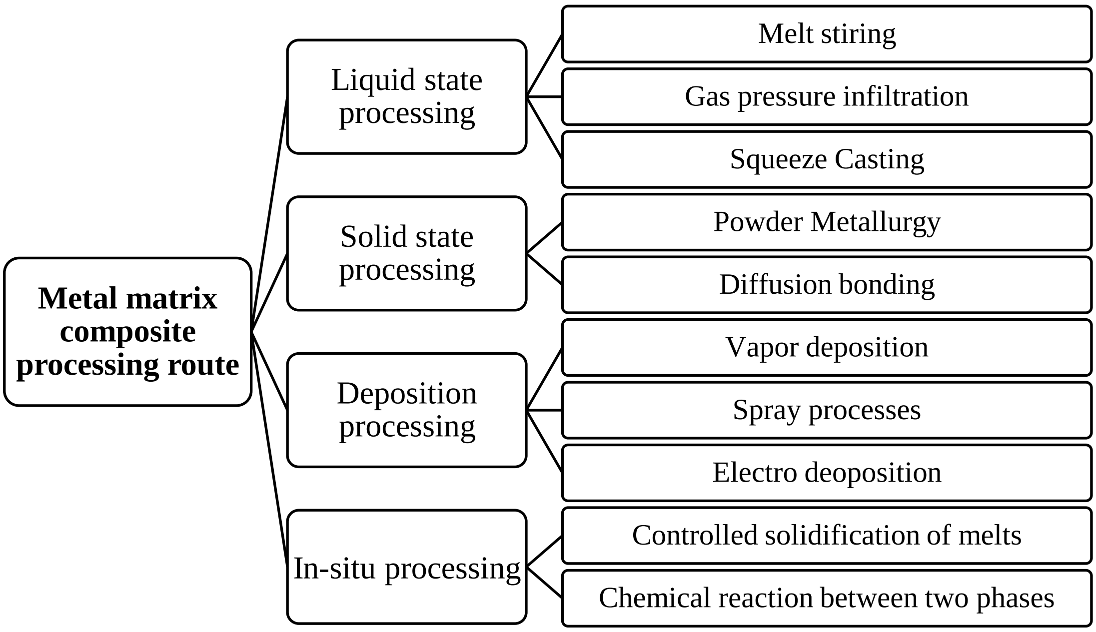

Fig. 2. 2 Classification of the processing routes to develop MMCs

2.2.2 Solid State Processing

The fabrication of the MMCs using solid-state processing involves reinforcement of metals with particulates without melting them. In this, the metal powders are blended with reinforcement particulates, compacted, and then consolidated through heat and pressure. It can be classified into two categories: powder metallurgy and diffusion bonding. Unlike liquid-state processing, solid-state techniques prevent issues related to high temperature degradation of the reinforcing phase and can produce composites with improved mechanical properties and uniform dispersion of reinforcements.

2.2.2.1 Powder metallurgy (P/M)

Powder metallurgy is a technique where the desired mixture of matrix and reinforcements in powder form is blended, compacted, and then heated (sintered) at elevated temperatures to initiate diffusion. Figure 2.3 illustrates the steps involved in the fabrication of composites via powder metallurgy (P/M) technique. This technique provides a flexible way to incorporate several kinds of reinforcement, such fibers, whiskers, or particles. The high versatility of the technique makes it possible to combine dissimilar materials that no other technique can do.

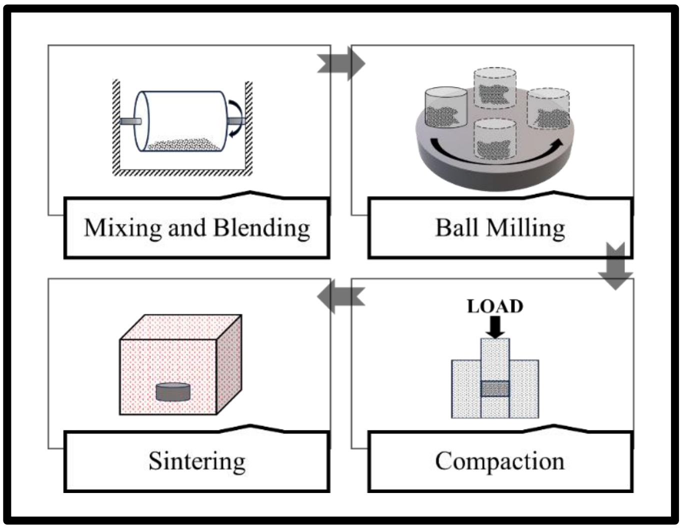

Fig. 2. 3 Steps involved in the powder metallurgy process

Mixing and Blending: The mixing is basically the incorporation of reinforcement materials such as ceramics, fibers, or other metal powders into the metal matrix in order to enhance properties such as strength, hardness, and tribological performance. The blending is done in order to get homogeneous distribution of all constituent phases. It also ensures consistent properties in the final composite. Since abrasive particles might harm the die surface, the lubricant is added to the powders to lessen wear and friction during compaction. Several factors need to be considered in mixing and blending: (a) milling time: excessive mixing can harden the particles and degrade the composite's quality, (b) milling speed: it should be optimized so that excessive heat generation and degradation of the powder can be avoided. while insufficient time may result in incomplete mixing, (c) ball to powder ratio: it affects the energy transfer efficiency, and a high value of ball to powder ratio increases the impact energy but can lead to excessive heating and contamination, (d) type and size of milling balls/vials: it should be harder than the material being milled to avoid contamination. Common materials include steel, tungsten carbide, zirconia, and alumina. The appropriate size of the ball/vial should be used as smaller ones provide higher surface area and more frequent impacts, while larger ones provide higher impact force, (e) process control agents (PCAs): prevent excessive cold welding and control particle size and shape, and (f) atmosphere: Inert gases (e.g., argon, nitrogen) may be used to prevent oxidation or contamination. A vacuum environment might be necessary for reactive materials. The planetary ball mill, which consists of a jar fixed to an eccentric sun wheel that rotates in opposite directions to efficiently coordinate the alternating centrifugal force, is a common tool used in mixing/blending processes to reduce the size of powder particles while also facilitating their thorough mixing.

Compaction: Compaction is basically the process of compacting the powdered mixture within a die by applying significant pressure under ambient conditions, causing green compact formation. It provides green strength to the compacts as it permits the particles to slide, interlock, and undergo plastic deformation. The compaction pressure is directly proportional to the compact density, while it is inversely proportional to the porosity of the compact [3].

Sintering: The process of sintering involves heating the green compact to assist the fusion of pores between the interlocking particles. The parameters such as temperature, pressure, holding time, and heating/cooling mainly govern the densification during the sintering. It involves three stages- (a) In the initial stage, interparticle welding occurs among loosely arranged particles, and the weld enlarges as the sintering time progresses, (b) In the intermediate stage: the network of pores becomes unstable and begins to contract, and (c) In the final stage: pores disappear [4]. Figure 2.4 illustrates the various sintering stages and the associated diffusion that occurs in the process.

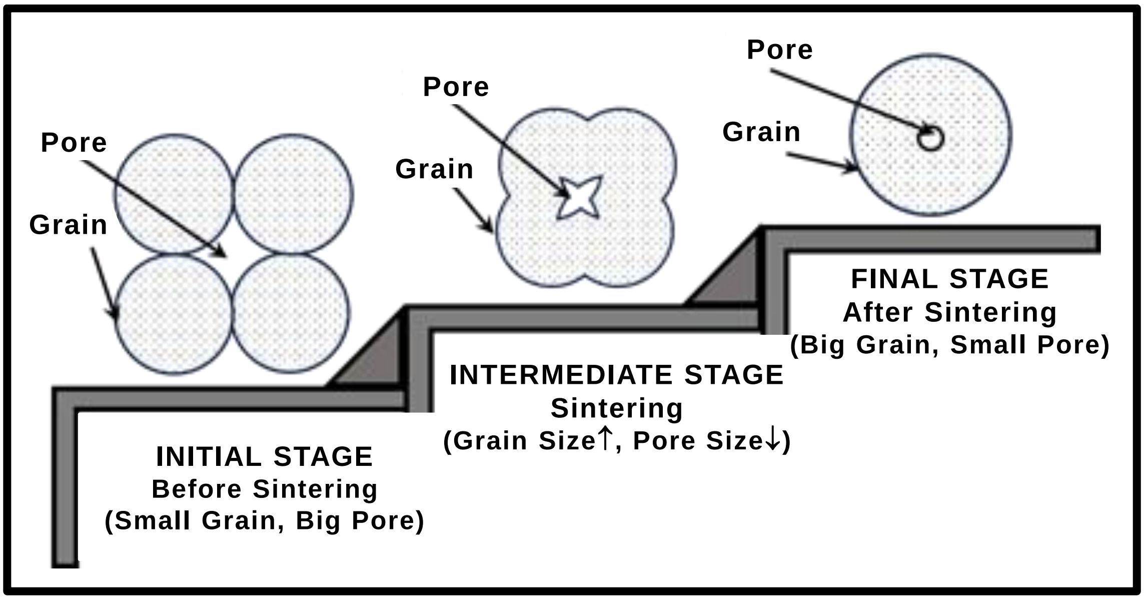

Fig. 2. 4 A schematic diagram of sintering

A number of sintering techniques, such as **conventional sintering, microwave sintering, hot press sintering (HP), hot isostatic pressing (HIP),** and **spark plasma sintering (SPS),** have been utilized in the fabrication of composite materials. The **conventional sintering process** is the simplest and most traditional process involving heating via conduction, convection, and radiation. In this slow heating rates and long dwell times during the sintering cycle often cause exaggerated grain growth phenomenon. **Microwave sintering** via volumetric heating mechanism enables a numerous advantage such as enhanced diffusion process, high heating rates, shorter processing times, and improved microstructure. **Hot pressing (HP)** involves simultaneous application of temperature and pressure resulting into a high-density composite compared to conventional sintering. If this sintering is performed under vacuum or inert atmosphere, then it is termed as vacuum hot press sintering. **Hot isostatic pressing (HIP)** involves the uniform application of pressure from all the directions via a gaseous/molten salt medium leading to uniform densification in all directions. **Spark plasma sintering (SPS)**, is a variant of hot pressing in which the heat source is a pulsed DC current that is passed through the die or the powders (depending on whether the powder is electrically conducting) during consolidation [5].

2.2.2.2 Diffusion bonding

Diffusion bonding, a solid-state processing, involves the embedding of fibers and metal sheets or foils alternately to create a sandwich structure. The obtained sandwich structure is compressed and heated at a temperature (more than the melting point of foil material), allowing the fiber to be wetted and the metal to spread throughout the structure.

2.2.2.3 Deposition processing

The deposition process can be classified into two main categories: **spray co-deposition** and **vapor deposition processes**. In **the Spray Co-Deposition process,** a high-speed cold, inert gas (argon or nitrogen) jet is used to fragment a stream of molten metal into droplets (300 μm or less). These fine droplets are sprayed with reinforcement particles and gathered on a substrate/ mould, which recombines these semisolid metal droplets and solidifies to produce the composite material. As the metal droplets impinge with very high velocity in a molten or semi-solidified state, they get flattened and welded together, forming a composite. It is known as a hybrid rapid solidification technique due to the rapid transformation of metal from the liquid state to the solid state, followed by a slower cooling to room temperature, as noted in [6,7]. **Electron Beam/Physical Vapour Deposition (EB-PVD)** involves heating of the target to the evaporation point via an electron beam in a vacuum chamber, and evaporated atoms originating from the target get deposited onto the substrate. This process enables condensation, resulting in the production of a relatively thick coating on the substrate [8,9]. Several types of evaporation sources can be used in this technology, which efficiently control the evaporation rate of various sources to enable composition modification. Vapour phase deposition has several benefits, including the ability to produce a wide range of compositions, the absence of mechanical disruption of the interfacial region, the ability to achieve uniform thickness, and the ability to manage thickness. At present, two techniques of vapour deposition are widely used such as physical vapour deposition (PVD) and chemical vapour deposition (CVD). **Physical vapour deposition (PVD)** involves the evaporation of the material via a high-energy source, such as an electron beam or ions from a source (known as a target) through the vapour phase to form a coating on a substrate. This technology involves techniques such as evaporation, ion plating, sputtering, and laser ablation. On the other hand, **chemical vapour deposition (CVD)** involves the deposition of a solid material from a gaseous phase through a chemical reaction between volatile precursors and the surface to be coated. As these precursor gases flow over the heated surface of the substrate, the chemical reaction produces a solid phase that deposits onto the substrate [10,11].

2.2.2.4 In-situ processing

The in-situ processing of the composites can be classified in two ways: (i) controlled **solidification of melts** [12–15] and (ii**) chemical reaction** between two phases [16–20]. The composites fabricated via this technique are relatively better compared to the above-mentioned techniques as they exhibit a homogenous distribution of reinforcing phases, and the spacing/ size of the reinforcing phases can be controlled by parameters such as solidification/reaction time. The in-situ crystallization of constituent phases leads to clean, coherent, and mutually compatible interfaces. However, its applicability is restricted due to the narrow range of matrix selection and reinforcements as well as the shape of the reinforcing phase and process kinetics system (in the case of reactions), is sometimes difficult to control [21].

2.2.3 Spark Plasma Sintering (SPS)

Spark plasma sintering, also known as pulsed electric current sintering (PECS), utilizes uniaxial force and pulsed direct electric current under a low atmospheric pressure/ inert atmosphere for the high-speed consolidation of the powder. The basic configuration of the spark plasma machine is shown in Fig. 2.5. It consists of a vertical single-axis pressurization mechanism, single/dual vertical loadings, single axis load frame, a press with a position measuring unit, a built-in water-cooled chamber, specially designed punch/electrode and vacuum chamber incorporated with a water cooler, temperature monitoring unit, DC pulse generator etc. The main working principle of SPS is based on the phenomenon of "Electro Spark Discharge," in which high energy and low voltage spark pulse current momentarily produces a spark plasma. This spark plasma results in a localized temperature of several thousand degrees between the particles, leading to optimum thermal and electrolytic diffusion. A number of researchers have illustrated the mechanism involved in SPS in the following ways: (i) Plasma generation, the generation of plasma discharge along with spark between the particles; (ii) Electro plastic effect, the plastic deformation of the metal particles under an electric field; (iii) Joule heating: the welding between the particle due to heat generated by passing of current, (iv) Pulsed current: the consolidation of the particle due to high amperage pulsed direct current, and (v) Mechanical pressure: the densification due to simultaneous application of pressure and temperature. Spark plasma sintering has several benefits compared to conventional systems (hot press (HP) sintering, hot isostatic pressing (HIP), and atmospheric furnaces), such as ease of operation, accurate control over sintering energy/speed, high reproducibility, safety, and reliability [22–24] .

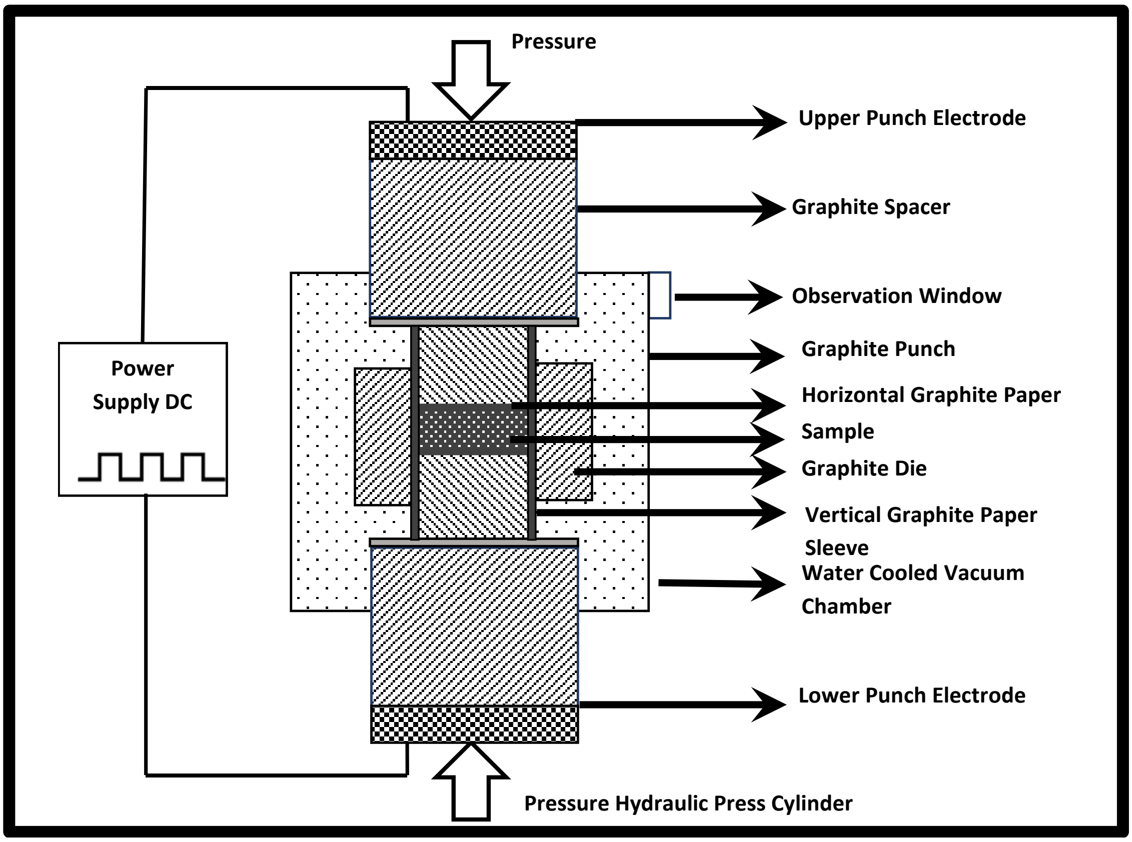

Fig. 2. 5 A schematic diagram of the Spark Plasma Sintering (SPS) machine

2.3 TRIBOLOGY

Tribology is the science and technology that basically deals with the analysis of the acceptable wear or lifetime of the parts of mechanical systems used in daily life as well as industries, having relative motion from the study of friction, wear, and lubrication. The tribology affects a number of industries as well as our everyday life in the following ways: (i) enhance machinery efficiency, (ii) reduce energy consumption, and (iii) prevent premature wear and failure [25]. Wear is associated with material wastage and loss of mechanical performance, and its reduction can lead to considerable savings. Friction is the primary source of wear and energy dissipation. Therefore, the reduction in friction results in considerable savings as $\sim 1/3 \ \ to \ \ 1/2$ of the world's energy is associated with friction [26]. Both friction and wear can be controlled effectively by means of lubrication. Lubrication can be done using gas, liquids, and solids, depending on the type of applicability. Therefore, the service time of a mechanical system can be enhanced by controlling either friction or wear [27]. The selection of materials/ lubricants and utilizing surface modification techniques are the major factors that are responsible for decreasing friction, minimizing wear, and preventing catastrophic failure in machine components.

2.4 WEAR AND ITS TYPES

Wear is an inevitable phenomenon during the sliding/slip of interacting surfaces. Due to the presence of hills and valleys on all the surfaces, the load is supported only at suitably oriented high regions of the interfaces. It leads to a much smaller apparent area of contact compared to the true area of contact, resulting in high contact stresses at the interacting surfaces. Based on factors such as the visual changes in worn parts or mechanisms and the specific conditions under which material is removed, wear can be classified as adhesive wear, abrasive wear, erosive wear, fatigue wear, fretting wear, and corrosive wear.

Adhesive wear is also known as sliding wear due to the important role of adhesion in sliding wear. When two solid surfaces come into direct contact during relative motion and adhere to each other, resulting in the transfer of material from one surface to another. The terms such as scuffing, scoring, and galling are often associated with severe sliding wear. *Scuffing* involves wear in the inadequately lubricated metallic contact during relative motion. *Scoring* involves the formation of grooves and scratches in the direction of sliding and can also indicate scratching by abrasive particles. *Galling* represents a more severe form of scuffing due to local welding leading to gross surface damage. **Abrasive wear** occurs in the presence of hard particles, either as a separate component between the sliding surfaces or as an element of the structure of one or both surfaces. In this type of wear, the material removal from the interacting surface having relative motion takes place due to the mechanical action of abrasive substances such as particles, grit, or abrasive tools. **Erosive wear** involves the material removal due to the impact of solid/liquid particles against the surface of an object. It occurs in a wide range of mechanical systems, and typical applications include damage to gas turbine blades by dust clouds on an aircraft while flying and wear in the impellers of the pump in mineral slurry processing systems. **Fatigue wear** is caused by deformations on the asperities and surface layers due to the interaction between asperities of opposing surfaces during relative motion. Repeated loading and unloading of high contact stresses caused by the contact between asperities during sliding/rolling lead to the generation of wear particles via fatigue-propagated cracks. The terms such as contact fatigue or surface fatigue are commonly used for surface damage caused by repeated rolling contact. **Fretting wear** is the material removal due to the small oscillatory movement (~1 to 300 μm) between the surface during motion. This oscillatory displacement increases the possibility of more damage caused by the wear debris in the contact area, which results in catastrophic failure. **Corrosive wear** is a general term relating to any form of wear dependent on a chemical or corrosive process. In this, surfaces having relative motion are exposed to corrosive substances like chemicals, moisture, or aggressive environments, resulting in the degradation of the material. If the corroding agent is atmospheric oxygen, then this wear is termed as *oxidative wear*.

2.5 FACTORS GOVERNING WEAR

Wear during motion depends on the sliding distance but also, to some extent, on sliding velocity and the test duration. The sliding velocity controls the dissipation rate of frictional energy and the temperature at the interface. Since abrupt transitions in wear mechanism and rate may occur with the change in the sliding speed, it is definitely not reasonable to assume that one wear test of half the duration at twice the sliding velocity will yield the same findings as another. Wear is also governed by the **nominal contact pressure** between the sliding surfaces and its transitions are commonly induced by changes in contact pressure. The **linear dimensions of the specimen** play an important role, independent of the contact pressure, as the presence of wear debris at the leading edge of a long specimen will have more influence during its passage through the contact zone than the shorter specimen. Other factors, such as test temperature, environment, orientation of apparatus, etc., also have a vital effect on the wear of the materials. The **testing temperature** is important through its influence on the mechanical properties of the materials and on thermally activated chemical processes. However, these may often be dominated by the rise of frictionally generated temperature. **Atmospheric composition** is exceptionally important because the reactive components, such as water vapour and oxygen, strongly influence wear rates and mechanisms in all types of material. The **orientation of the apparatus** affects the outcomes of the testing as different behaviour may be seen if wear debris falls readily away from the contact area under gravity rather than being retained in the counterface. After a number of experiments for many systems, the loss of material by wear is found to be directly proportional to the sliding distance (and so, to the time during sliding at a constant velocity).

According to Archard [28], the wear rate of material during sliding is proportional to the distance slid. Sometime, the transient behaviour can be seen from the start of sliding to the equilibrium surface conditions. During the initial running period, the wear rate of the material may be either higher or lower compared to steady state wear rate depending on the nature of running-in process, so, this proportionality is less often found. The variation in the sliding conditions such normal load, sliding velocity, and in some cases sliding time/distance are responsible for the transitions in the dominant wear mechanism and in the associated rate of wear. Consideration of the parameters such as **mechanical stresses, temperature,** and **oxidation phenomena** is necessary in understanding the wear of metals during sliding as the different conditions at the interface especially temperature from those in the surroundings. The understanding of the sliding wear turns out to be difficult as all three controlling factors are interrelated and may be influenced by both load and sliding velocity. An increase in the load results into higher stresses and more severe mechanical damage. The load as well as sliding velocity affect the interface temperature due their control on the power dissipated at the interface (since the power dissipation is the product of the sliding speed and the frictional force). Furthermore, the sliding velocity also determines the relative importance of heat conduction away from the interface. The low sliding velocity is responsible for relatively rapid conduction of the heat generated during sliding leading to low temperature at interface, and the limiting conditions would be isothermal. The high sliding velocity assists only limited heat conduction at the interface temperature resulting into high temperature at interface, and the limiting conditions would be adiabatic. The high temperature at the interface results into high chemical reactivity of the surfaces which causes rapid growth of oxide films in air. The oxide films have a complex and context-dependent impact on wear, whether or not they contain lubricious compounds. Oxide films generally serve as protective barriers that reduce wear by reducing friction and preventing direct contact between surfaces. However, specific circumstances, such as high temperatures, may lead to the breakdown of oxide layers, potentially increasing wear. The overall impact is influenced by the factors such as material type, environment, and load, making a deep understanding of these conditions essential for accurate predictions.

2.6 LUBRICATION

The main purpose of lubrication is to control friction and minimize wear by forming a thin protective film that reduces the friction between interacting surfaces in motion. Another substantial benefit of lubrication is the reduction in the rate of sliding wear to a greater/lesser extent. Several lubrication mechanisms, such as liquid, solid, semi-solid, and gas lubrication, can be utilized to tackle the diverse challenges faced in the advancement of technology. The selection of type of lubricant depends on the applicability in the environment type such as vacuum, cryogenic, high temperature, and radiation as illustrated in Fig. 2.6.

Hydrodynamic lubrication involves the separation of the sliding surface by a fluid film, which is comparatively thicker than asperity heights in the bearing surfaces. The hydrostatic pressure in the film produces only small elastic distortion of the surfaces, which can be treated as rigid. **Elasto-hydrodynamic lubrication** is similar to hydrodynamic lubrication. Elastic deformation of the surfaces can no longer be neglected in this type of lubrication because of the extremely high local pressures and thin lubricant coating; in fact, it is an essential component of this lubrication regime. **Boundary lubrication** is a type of lubrication where surfaces are separated by absorbed molecular films usually laid down from oil or grease comprising an appropriate boundary lubricant; appreciable asperity contact, and junction contact may nevertheless occur. Liquid lubricants restrict their applicability due to following factors such as prone to contamination in the corrosive environment and decomposition or oxidation at a high temperature, which leads to a decrease in viscosity and deterioration in the performance. They may not be suitable due to challenge in sealing issues, weight constraints, or other factors.

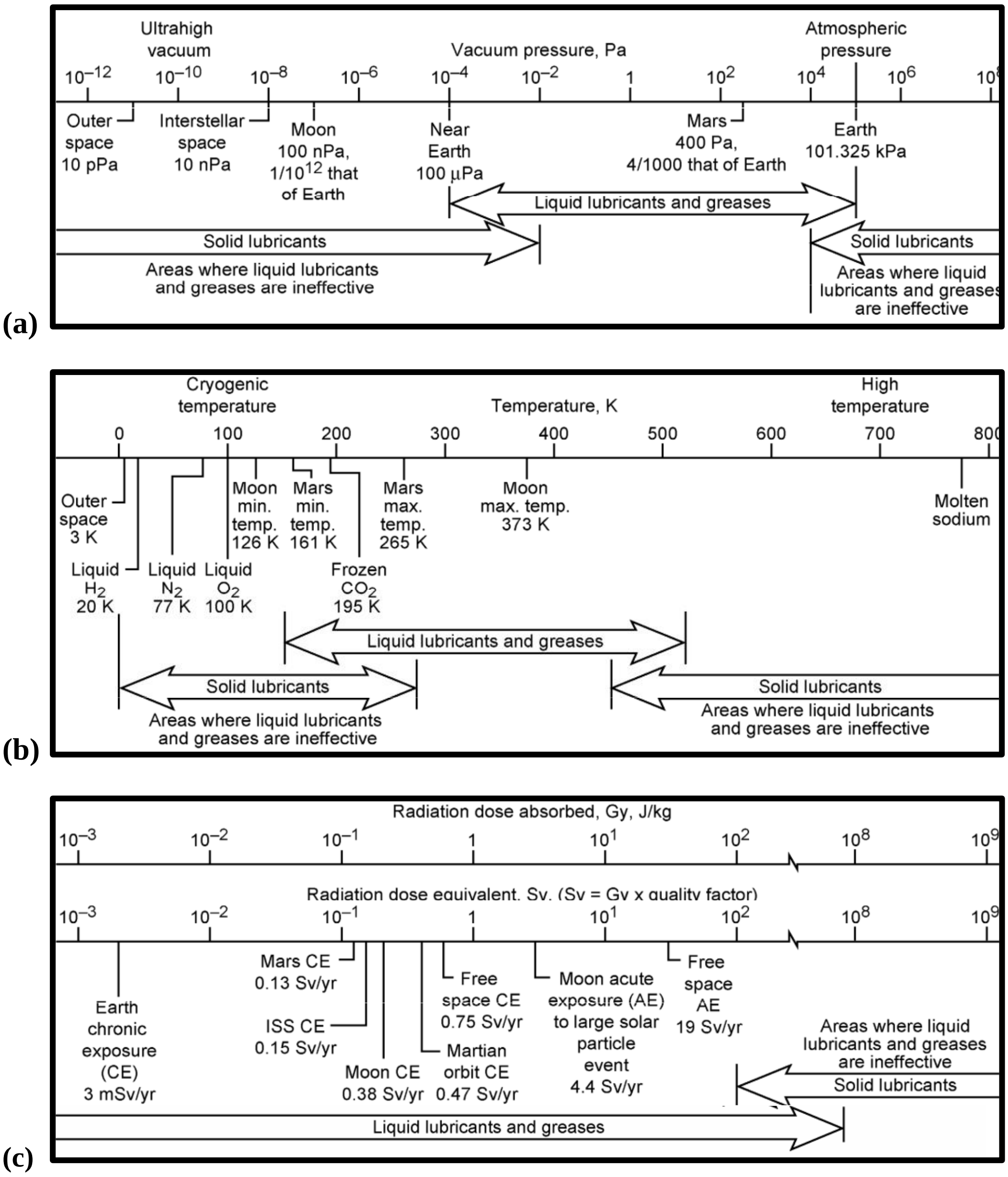

Fig. 2. 6 Ranges of application of various lubricants in (a) vacuum environments, (b) cryogenic and high temperature environments, and (c) radiation environments [29]

Solid lubrication has been done where gas and liquid are not able to able provide lubrication efficiently. The interaction surfaces exhibit considerable adhesion during the relative motion in the absence of lubrication provided by liquids or gases. This adhesion in between rubbing surfaces nearly always results in high friction as most materials resist shear parallel to the surface of interaction as efficiently as they resist compression normal to the contact face. Though, the failure of anisotropic materials takes place at low shear stresses, leading to low friction at the interface. The characteristics of lamellar solid are anisotropy of mechanical properties, or in simple terms, planes of weakness. The sliding ability of the lamellae over one another at relatively low shear stresses makes a lamellar solid to self-lubricating material. Few non-lamellar solids such as silver can also decrease both friction and wear on the depositing a thin film onto the interacting surfaces. Hence, a second mechanism of solid lubrication is the film deposition of soft metals on a hard substrate [30]. The hardness of the substrate controls the contact area and no matter how thin the soft metallic layer is, the shear strength of asperities in contact is determined by the softer and weaker metal. Consequently, the product of asperity shear strength and contact area which determines frictional force becomes quite low under such conditions. Therefore, lamellar solids and soft films provide the two fundamental modes of solid lubrication currently employed.

The solid lubricants are needed when liquid lubricants do not meet the advanced requirements such high vacuum (such as in space), high temperatures, cryogenic temperatures, radiation, dust, clean environments, or corrosive environments, and combinations thereof. The materials designed for solid lubrication must not only display desirable coefficients of friction (0.001 to 0.3) but must maintain good durability in different environments, such as high vacuum, water, the atmosphere, cryogenic temperatures, high temperatures, or dust. Solid lubricants such as soft metals, transitional metal dichalcogenide compounds(TMDs), polymers, soft metals, metal fluorides and oxides, hexagonal boron nitride (*h*-BN), and graphite/its derivatives are widely employed as solid lubricant.

Soft metals such as silver (Ag), tin (Sn), gold (Au), lead (Pb), and indium (In) etc., have crystal structures with multiple slip planes and they do not work harden significantly. The frictional heat generated during sliding rapidly diminishes the dislocations and point defects generated during shear deformation [31] and sliding contact. are soft enough to act as solid lubricants [32]. The inherent properties of the soft metals like low surface roughness and high viscosity are responsible for their distinctive characteristics. The heat generated through friction during sliding of soft metals serves to eliminate lattice defects, like dislocations and vacancies [33]. This elimination of lattice defects results in improper work hardening, which is the key factor contributing to superior lubrication in challenging conditions. There is a widespread use of Pb, In, and Sn in bearing alloys including Babbit metals based on needle shaped intermetallic Sn-rich matrix, leaded bronzes containing islands of Pb in a bronze matrix, Al-Sn alloys. Thick overlays are applied in steel backings during the fabrication of the bearings [34]. Based on low hardness and oxidation characteristics, only Ag, Au and their alloys seem to offer lubrication at higher temperatures. Silver is the ideal choice among soft metals for self-lubricating materials in challenging conditions, primarily due to its superior oxidation resistance and high thermal conductivity, enabling efficient dissipation of heat generated during relative motion. The primary self-lubrication mechanism of silver is its exceptional diffusion and the facile formation of a tribo-layer at the interface. Consequently, silver is capable of delivering superior lubrication properties when operating at temperatures below 500 ℃ [35].

Transition Metal Dichalcogenides (TMDs), such as sulphides and diselenides of Mo and W exhibit closely packed lamellar structure with strong bonding between metal and chalcogenide ligands, which differs with the weak van der Waal adhesion forces between sulphur like atoms leading to the formation of easy-to-shear lamellas. Additionally, they can also align in the sliding direction, lead to reduce friction of the tribo-system

[33,36]. **Metal oxides** such as PbO, MoO_3, Cr_2O_3, and NiO, etc., are effective solid lubricant at high temperature due to their low shear strength at elevated temperatures. They do not possess lubricating properties at room temperature (RT). Many oxides exhibit a change in tribological behaviour as the temperature increases. The observed changes in tribological properties can be attributed to the transition from brittle to ductile behaviour, which occurs when the temperature exceeds a critical threshold. Hence, temperature significantly influences oxide lubrication [33,37].

Fluorides of metals (BaF_2, CaF_2, and LiF) and rare earth elements (CeF_3 and LaF_3) and LiF exhibit significantly better tribological properties above 500 °C due to transition from brittle to ductile wear mechanism. The high friction and increased wear rate at low temperature are credited to three-body abrasion. CaF_2 and BaF_2, being soft, poorly soluble in water, and thermally stable across a wide temperature range, have a thermal expansion coefficient that closely matches that of many alloys [33,37,38].

Hexagonal boron nitride (*h*-BN) has been reported as a clean and high temperature solid lubricant due to its lamellar structure powder like graphene, MoS_2 etc., high thermal stability, good chemical inertness and high thermal conductivity at elevated temperatures i.e., above 500 °C. However, its applicability is restricted due to the poor sintering and wetting characteristics in various metal matrices [39–41]. Recently, a couple of studies have shown that these features of *h*-BN can be improved by modifying the *h*-BN powder with Cu or Ni [42–44]. Shi et al.[41] fabricated Ni_3Al based self-lubricating composites containing 0, 10, 15, and 20 wt.% of WAh (i.e., of WS_2, Ag, and *h*-BN with a weight ratio of 1:1:1) via spark plasma sintering and observed that the composite containing 15 wt.% WAh had the lowest coefficient of friction(CoF) and wear rate in comparison to other composites under a constant load of 10 N and fixed sliding speed of 0.234 m/s. Mahto et al.[42] also reported improved tribological performance in terms of CoF and wear rate for Ni_3Al based composites containing 10 wt.% Ag and 5 wt.% Cu-*h*-BN in comparison to single solid lubricants (Ag /Cu-*h*-BN). In addition, Tyagi et al.[39] found that a combination of solid lubricants revealed improved tribo-performance than a single lubricant at a fixed sliding speed of 1.0 m/s and a constant load of 20 N under dry sliding wear against an AISI2100 steel pin from room temperature to 600 ºC. Hence, it becomes imperative to modify *h*-BN with either Cu or Ni to enhance its wetting characteristics as Cu and Ni are mutually soluble.

Polymers like polyimide (PI), polytetrafluoroethylene (PTFE), and polyetheretherketone (PEEK) act as excellent additive for solid lubrication due to their low densities, good corrosion resistance, good machinability, and able work under vacuum and at cryogenic temperatures [45,46]. Their applicability is restricted due to excessive flow under load (specifically at elevated temperatures), low thermal conductivity, high thermal expansion, low radiation stability, low heat dissipation efficiency, and lack of strength and dimensional stability [37].

Graphene and its derivatives such as few-layer graphene, multilayer graphene (MLG), graphene oxide (GO), reduced graphene oxide (rGO), functionalized graphene and so on, has emerged as promising solid lubricant at nano-, micro-, and macro- scales due to excellent mechanical strength, thermal stability, low surface energy, and atomic smooth surface of graphene. Reduced graphene oxide (rGO), being a graphene derivate, has also emerged as a potential solid lubricant due to its low preparation cost compared to graphene and similar mechanical and tribological properties to graphene [47]. The presence of oxygen-containing groups such as epoxy and hydroxyl groups in rGO causes increased adhesion and shear strength between the graphene layer, leading to higher friction compared to graphene [48]. Numerous studies [47,49–52] conducted on rGO as an additive in lubricants/coatings have reported an improvement in their tribological performance. A limited number of investigations have been performed on rGO as a solid lubricant reinforcement in metal/intermetallic-based composites. Nie et al. [53] prepared Cu composite with Cu modified rGO via one step reduction with modified molecular level mixing method and hot press sintering, and found low CoF and low wear rate at RT sliding against steel ball compared to pure Cu. Zhang et. al [54] fabricated Cu based composite with hybrid of short carbon fiber-rGO and reported improved tribological performance at RT against steel compared to both pure Cu as well as Cu-rGO composite. Nautiyal et. al [55] also reported a reduction in CoF and wear rate of Cu composite after reinforcing with hybrid rGO-MoS_2 at RT in comparison to pure Cu and composites containing either rGO or MoS_2 alone. Liu et. al [56] also reported an improvement in tribological performance of the Al composite having chemically modified multilayer graphene oxide (mrGO) by Cu after sliding against steel at RT. The intermetallic (Fe–Ni) reinforced with hybrid material (MoS_2/rGO) exhibited improved tribological performance against an Al_2O_3 ball from RT to 600 °C [57]. It has been indicated that the poor wettability of rGO with several metal-based matrices restricts its applicability as a solid lubricant in metal matrix composites [53–57]. Hence, there is a need to modify rGO to enhance its wettability with the metal matrix.

2.7 NEED FOR SOLID LUBRICATION AT ELEVATED TEMPERATURE

Advancements in science and technology have driven the need for materials for the components of industrial tribo-systems, capable of providing effective lubrication in an extended range of high temperatures, loads, speeds, and highly reactive environments. The potential application areas include space satellites, Stirling engine cylinders, rotating face valves, high-speed foil air bearings, and hypersonic aircraft and missiles [33,37]. As per the requirements of the high technology areas, the use of solid lubricants (SLs) is the only viable alternative to reduce friction under conditions such as (i) **Extreme Temperature**

and Pressure Conditions where other lubricants fail to work effectively, i.e., at extremely low temperatures down to -212 ^{0}C, where lubricants may solidify; high to full vacuum applications such as aerospace where lubricants may volatilize, (ii) **Intermittent Loading Conditions**, i.e., if an equipment is stored or idle for prolonged periods, solid lubricants provide permanent noncorrosive lubrication, (iii) **Inaccessible Locations** which are difficult to servicing, solid lubricants offer special advantage, if selected properly for the intended load and speed application, (iv) **High Dust and Lint Areas**, where fluids will tend to pick up dust and lint with liquid lubricants, these may form a grinding paste and that may cause damage to equipment, (v) **Contamination i.e.,** solid lubricants may be used where liquid lubricants have to migrate to other location and cause contamination of other equipment's, parts or product, and (vi) **Environment** where the lubricated equipment is immersed in water that may be polluted by other lubricants such as oils and greases.

2.8 SELF-LUBRICATING MATERIALS FOR HIGH TEMPERATURE APPLICATIONS

The fabrication of the self-lubricating materials for application at high temperature is a complex and challenging task as it requires a multidisciplinary approach including chemistry, physics, metallurgy and surface engineering, etc. A number of requirements has to be fulfilled by the self-lubricating materials to perform satisfactorily at high temperatures which are as follows: (i) low shearing ability of the active solid lubricant in the direction of sliding by means of mechanisms either increased ductility at elevated temperature or easy -to-shear lamellar microstructure or melting, (ii) mechanical strength, thermal stability, oxidation and/or oxidation resistance, (iii) controlled diffusion toward the contact region in order to avoid accelerated depletion of solid lubricants, (iv) high thermal conductivity of the resulting material for dissipating frictional hearting from contact zone, and (v) non-toxic and environment compatible.

The matrix material and solid lubricant are required to be mutually compatible to provide low co-efficient of friction and high wear resistance at elevated temperatures. Additionally, composite should have adequate hardness as well as ductility and high oxidation resistance at high temperature to avoid oxidative wear. Furthermore, it should possess appropriate thermal coefficient of expansion so that lubricating film can be formed on the worn surface. The metals, intermetallic compounds, and ceramics are the appropriate materials to be used as a high temperature matrix material [33]. Superalloys, usually based on Ni, Fe, or Co are widely used as at high temperature applications due to their exceptional corrosion and oxidation resistance properties and ability to retain their stiffness, strength, toughness and dimensional stability at temperatures much higher than the other aerospace structural materials. Among these, Nickel based superalloys exceptional group of superalloys as they have high creep resistance at high temperatures (850 °C). Ni superalloy can be alloyed with various elements such Cr (5-15 wt.%) for oxidation and hot corrosion resistance, carbides, and solution hardening, Mo and W (0-12 wt.%) for carbides and solution hardening, Al (0-6 wt.% wt.%) for precipitation hardening, oxidation resistance and γ’ former, Ti (0-6 wt.% wt.%) for precipitation hardening, carbides and γ’ former, Nb (0-5 wt.%) for carbides , solution hardening, and precipitation hardening, and Ta (0-12 wt.%) for carbides, solution hardening, oxidation resistance and γ’ former [58].

Numerous studies are available in published literature on fabrication and tribological behaviour of Ni based self-lubricating composites at different loads, speeds, environment and temperatures against various counterface materials. Table 2.1 presents a comprehensive overview of these studies.

[Symbols used: L- Load, V- Sliding speed, t-Test duration, T-Test temperature, C-Counter face]

Table 2. 1 Review of the tribological properties of Ni-based high temperature self-lubricating composites

Author(s), Journal (Year)	**Fabrication/ Material used**	**Tribometer/ Testing Parameters/ Counterface**	**Results/Observations**
Dangsheng et al. [59] Wear (2001)	Hot Press Sintering NiCr- (0, 2, 4, 6, 10, 15, 20 wt.%) MoS_2	Pin on disc (Rotary) L = 49, 98, 147, 196 N; V = 2.5 m/s; t = 16.67 min; T = RT, 200, 400, 600 ^oC; C(disc) = High-speed steel	- The composite with 10 wt.% of MoS_2 showed the best combination of mechanical and tribological properties. - The glaze and oxides layer formed on the worn surface of 10 wt.% MoS_2 alloy at high temperature (> 600 °C) helped in reducing friction and wear.
Liang Li et al. [60] Wear (2008)	Hot Press Sintering NiCrWFeSiC- (0, 5, 10, 15 wt.%) MoS_2	Ball/Pin on disc (Rotary) L = 50 N; V= 0.8 m/s; t = 15 min (for RT, 200, 400 ^oC) and 5 min (600 ^oC); T = RT, 200, 400, 600 ^oC; C(ball/pin) = ball (Si_3N_4, Al_2O_3), pin (High-speed steel)	- The CoF (~0.27-0.14) and wear rate (~$1.0\text{-}3.5 \times 10^{-6}$ $mm^3/(Nm)$) were obtained for the composite having MoS_2 when rubbed against Si_3N_4 from RT to 600 °C due to a synergetic lubricating action of graphite and MoS_2. - With the increase of MoS_2 content, friction and wear varied slightly at low temperature, while the decreased at elevated temperature, it has been attributed to the presence of MoS_2 which helped in friction reduction at 600 ^oC.
Li et al.[61] Wear (2009)	Hot Press Sintering NiCr- (5, 10 wt.%) Ag- (0, 10 wt.%) MoS_2- (2, 4, 8 wt.%) CeO_2	Ball/ Pin on disc (Rotary) L= 20 N from RT to 600 °C; V = 0.4 m/s; t = 25 min; T= RT, 200, 400, 600 ^oC; C(ball) = Al_2O_3; C(pin) = High-speed steel, Ti_2AlNb	- The friction reduced from RT to 400 °C with the addition of Ag in the Ni-base composite. The addition of CeO_2 caused the formation of a smooth glaze layer during high temperature friction, reduced friction and wear at 600 °C. - The lowest CoF (~0.5-0.2) and wear rate ($<12\times10^{-5}$ mm^3/Nm) were obtained for Ni-based composite containing Ag-MoS_2-CeO_2 when slid against Ti_2AlNb compared to HSS and alumina.

Tyagi et al. [39] Wear (2010)	Hot Press Sintering NiCrWMoAlTi-(0, 8 wt.%) *h*-BN-(8, 12, 16, 20 wt.%) nano Ag	Ring on disc (Rotary) L= 20 N; V= 1.0 m/s; t = 10.42 min; T= RT, 200, 400, 600 °C; C(ring) = AISI52100 steel	• Composite having 8 wt. % *h*-BN and 12 wt. % Ag exhibited the lowest CoF due to the presence of Ag and *h*-BN on the worn surface of the composites. • Ag and *h*-BN were responsible for low CoF and wear rate from RT to 400 ^{o}C, whereas *h*-BN and lubricious oxide (WO_3, MoO_3, and $NiMoO_4$) for reduced CoF at 600 ^{o}C.
Li et al. [62] Tribol Lett (2013)	Hot Press Sintering NiCrMoAl -12 wt.% Ag -10 wt.% CaF_2/BaF_2	Pin on disc (Rotary) L = 10 N; V = 1.0 m/s; t = 20 min; T = 25, 200, 400, 600, 800 ^{o}C; C(pin) = Inconel 718	• The composite exhibited lower CoF (~0.25) from 25 to 800 °C due to supportive lubricating action between Ag and CaF_2/BaF_2. • Ag was responsible for lubrication at low temperatures (25-500 ^{o}C), whereas CaF_2/ BaF_2 eutectic at high temperatures (>500 ^{o}C).
Liu et al. [63] Tribol Lett (2012)	Hot Press Sintering NiMoAl-20wt.% Cr_2O_3-(0, 3.5, 5, 7.5 wt.%) graphite- 20 wt.% Ag_2MoO_4	Pin on disc (Rotary) L = 2 N; V = 0.287 m/s; t = 60 min; T = 20, 200, 300, 500, 700 ^{o}C; C(Disc) = Inconel 718	• The composite containing 7.5 wt.% graphite showed CoF (~0.25) and wear rate (~9.37 ×10^{-7} mm^3/Nm)) at 700 ^{o}C due to the synergistic action of dual lubricants.
Liu et al. [64] Tribology Transactions (2013)	Hot press sintering NiMoAl-20 wt.% Cr_2O_3- (0, 10, 20 wt.%) $Ag_2Mo_2O_7$	Pin on disc (Rotary) L = 2 N; V = 0.287 m/s; t = 60 min; T = 20, 200, 300, 500, 700 ^{o}C; C(disc) = Inconel 718	• Composite with 20 wt.% $Ag_2Mo_2O_7$ exhibited lower CoF (~0.33) and wear rate (~1.51 ×10^{-5} mm^3/Nm) and due to synergistic lubricating effect MoO_3, $Ag_2Mo_2O_7$ and iron oxide (Fe_2O_3 and Fe_3O_4) at 700 ^{o}C. • Ag acted as a lubricant at low temperatures (<500 ^{o}C), whereas molybdates and oxide provided lubrication at high temperatures (>500 ^{o}C).
Ouyang et al. [65]	HPS NiCr- (0, 10, 20,	Ball on disc (Rotary) L= 5 N; V = 0.126 m/s; t =	• The composite having 20 wt.% $BaCrO_4$ showed in reduction of CoF compared to NiCr alloy form 400-600 ^{o}C due to the formation of a

Wear (2013)	30wt.%) $BaCr_2O_4$	60 min; T= RT, 400, 600 °C; C(ball) = Al_2O_3	transfer containing different oxides (Cr_2O_3, NiO, $Ni_2Cr_2O_4$) and at 800 °C due to a protective oxide glaze layer composed of $BaCr_2O_4$ and $BaCrO_4$.
Ouyang et al. [66] Wear (2015)	Hot Press Sintering NiCr- (0, 10, 20, 30 wt.%) $BaMoO_4$	Ball on disc (Rotary) L= 5 N; V = 0.126 m/s; t = 60 min; T= RT, 400, 600 °C; C(ball) = Si_3N_4	• The composites exhibited inferior tribological properties compared to the base alloy at RT due to their brittle fracture. • NiCr-$BaMoO_4$ composites exhibited lower CoF (~0.30-0.26) and wear rate (~10^{-5}-10^{-6} mm^3/Nm) at elevated temperatures due to the combined lubricating action of $BaMoO_4$, $NiCr_2O_4$, NiO, and Cr_2O_3.
Wang et al.[67] Tribol Lett (2015)	Hot Press Sintering NiCr-(0,10 wt.%) Ag-(10, 0 wt.%) Mo/MoO_3	Ball on disc (Rotary) L= 20 N; V = 0.108 m/s; t = 60 min; T= RT, 300, 500, 700 °C; C(ball) = Al_2O_3	• Lowest CoF (~ 0.23) and wear rate (~1.35×10^{-5} mm^3/Nm) were observed for composite containing a combination of Ag and Mo. • Synergistic action of Ag_2MoO_4, $NiCr_2O_4$, and NiO improved the friction and wear performance at high temperatures.
Zhen et al. [68] Tribol Int (2014)	Hot Press Sintering NiCrMoTiAl-12.5 wt.% Ag- (5, 10, 15 wt.%) BaF_2 /CaF_2	Ball/pin on disc (Rotary) L= 5 N; V = 1.0 m/s; t = 30 min; T= RT, 200, 400, 600, 800, 900 °C; C(ball/pin) = ball (Si_3N_4), pin (Inconel 718)	• All Composites exhibited lower CoF and wear rate from RT to 900 °C sliding against Si_3N_4 compared to Inconel 718. • CoF of the composites remained almost same irrespective of the amount of BaF_2 /CaF_2 eutectic, whereas the wear rate increased with an increase of amount of BaF_2/ CaF_2 eutectic. • Ag provided lubrication below 400 °C, whereas at 600 °C Ag_2MoO_4, Cr_2O_3, and $BaTiO_3$, and at 800 and 900 °C $BaMoO_4$, $CaMoO_4$ were responsible for lubrication.
Zhen et al. [69] Tribol Int (2017)	Hot Press Sintering NiCrMoTiAl-20 wt.% MoS_2- (0, 12.5 wt.%) Ag-	Ball on disc (Rotary) L= 5 N; V = 1.0 m/s; t = 30 min; T= RT, 200, 400, 500, 600, 700 °C; C(ball) = Si_3N_4; Atmosphere = Air	• Addition of Ag and MoS_2 decreased the friction and wear of the composite from low to moderate temperatures, while the synergistic action of MoS_2 and CaF_2 resulted in friction reduction from moderate to high temperatures. • Composite having 20 wt.% MoS_2 and 5 wt.% CaF_2 exhibited the

	(0, 5 wt.%) CaF_2		lowest CoF (~ 0.40-0.16) and lowest wear rate (~1.0-29.4 ×10^{-5} mm^3/Nm)
Zhen et al.[70] Tribol Int (2017)	Hot Press Sintering NiCrMoTiAl-12.5 wt.% Ag-5 wt.% CaF_2/BaF_2	Ball on disc (Rotary) L= 5 N; V = 0.8 m/s; t = 30 min; T= 25, 200, 400, 600, 800 °C; C(ball) = Si_3N_4; Atmosphere = Air and vacuum	• The formation of Ag film from RT to 600 °C, resulted in reduced friction and wear of the composite in vacuum compared to that in air due to the oxidation of Ag film. • At 800 °C, a compacted glazed layer containing MoO_3, NiO, $BaTiO_3$, $BaMoO_4$, and $NiCr_2O_4$ decreased friction and wear in the air compared to that in vacuum.
Zhen et al.[71] Tribol Int (2022)	Hot Press Sintering NiCrMoTiAl-(0, 5, 10, 15 wt.%) MoS_2-(0, 5 wt.%) CaF_2	Ball on disc (Rotary) L= 5 N; V = 1.0 m/s; t = 30 min; T= 25, 200, 400, 500, 600, 700 °C; C(ball) = Si_3N_4	• Composites containing more than 5 wt.% MoS_2 showed superior lubricating behaviour compared to others. • Synergistic action of MoS_2, CaF_2 and the in-situ formed oxide reduced CoF and wear rate above 200 °C.
Zhen et al.[72] Lubricants (2023)	Hot Press Sintering NiCrMoTiAl-(10, 0, 10 wt.%) Ag_2MoO_4- (10, 10, 0 wt.%) $BaMoO_4$	Ball on disc (Rotary) L= 5 N; V = 1.0 m/s; t = 30 min; T= 25, 200, 400, 500, 600, 700 °C; C(ball) = Si_3N_4	• Composites containing a combination of Ag_2MoO_4 and $BaMoO_4$ exhibited the best lubricating properties from 400 to 800 °C, and CoFs and wear rates were ~0.50-0.34 and ~ 1.25-0.47 ×10^{-4} mm^3/Nm, respectively.
Zhen et al.[73] Lubricants (2024)	Hot Press Sintering Ni alloy- 12.5 wt.% Ag- 5 wt.% CaF_2/ BaF_2, (0, 5, 8, 12	Ball on disc (Rotary) L= 5, 10 N; V = 1.0 m/s; t = 30 min; T= 25, 200, 400, 600, 800 °C; C(ball) = Si_3N_4; Atmosphere = Air	• The composite containing 12 wt. % Mo exhibited the lowest CoF. • Ag provided lubrication from 25 to 400 °C, whereas the formation of an oxidative glazed layer was responsible for lubrication at 600 and 800 °C.

	wt.%) Mo		
Liu et al. [74] Tribology Lett (2013)	Hot Press Sintering NiCr-Al_2O_3- (0, 10 wt.%) $SrSO_4$- (0, 10, 15 wt.%) Ag	Ball on disc (Rotary) L= 10 N; V = 0.1 m/s; t = 60 min; T= RT, 200, 400, 600, 800, 1000 °C; C(ball) = Al_2O_3	• Composite containing 10 wt.% Ag and 10 wt.% $SrSO_4$ exhibited CoF (~0.48-0.28) and wear rate (~3.19 ×10^{-5}-5.46 ×10^{-6} mm^3/Nm) from RT to 1000 °C • The formation of a continuous lubricating film $SrAl_4O_7$, Ag, and $NiCr_2O_4$ from room temperature to 800 °C, and $SrCrO_4$, $NiAl_2O_4$, $NiCr_2O_4$, and Ag at 1,000°C resulted in a reduced CoF as well as wear rate.
Cheng at al. [75] Materials and Design (2017)	Hot Press Sintering NiCrMoAl-12.5 wt.% Ag- (5, 10 wt.%) CaF_2/BaF_2	Ball on disc (Rotary) L= 5 N; V = 0.785 m/s; t= 30 min; T= RT, 200, 400, 600, 800 °C; C(ball)= Si_3N_4; Atmosphere = Air and Vacuum	• The formation of Ag film below 400 °C and synergistic effect of Ag and fluorides at 600 °C helped in reducing friction. However, BaF_2/CaF_2 provided lubrication at 800 °C, irrespective of the environment. • The wear rate of the composites approximately doubled (in magnitude) in air than in vacuum due to surface oxidation.
Feng et al. [76] J. Wuhan Univ. Technol.-Mat. Sci. Edit (2019)	Spark Plasma Sintering NiCr-20 wt.% Ti_3SiC_2- (0, 3, 5, 10 wt.%) $Ag_2W_2O_7$	Ball on disc (Rotary) L = 10 N; V = 0.2 m/s; t = 60 min; T= 20, 200, 400, 600 °C; C(ball) = Si_3N_4; Atmosphere=Air	• Composite with 20 wt.% Ti_3SiC_2-5wt.% $Ag_2W_2O_7$ exhibited the lowest friction (~0.49-0.33) and wear rate (~7.07-9.89 ×10^{-5} mm^3/Nm) over a wide temperature range due to synergistic effect of Ag and lubricious oxides of Ni, Ti, Si, and W.
Li et al. [77] Wear (2021	Hot Press Sintering NiCr-10 wt.% Mo- (0, 10, 20, 30 wt.%) Bi_2O_3/TiO_2	Ball on disc (Rotary) L = 10 N; V = 0.104 m/s; t = 30 min; T= 800 °C; C(ball) = Al_2O_3; Atmosphere = Air	• The composite with 20 wt.% Bi_2O_3/TiO_2 showed lowest wear rate of ~2.1 × 10^{-5} mm^3/Nm and improved tribological performance due to formation of $Bi_4Ti_3O_{12}$ on the worn surfaces.

Shan et al.[78] Tribol Lett (2023)	Hot Press Sintering Ni18Cr9W-10 wt.% Al_2O_3- (0, 5 wt.%) $SrCO_3$- (0, 5 wt.%) Ag	Pin on disc (Rotary) L= 10 N; V = 0.5 m/s; t = 120 min; T= RT, 200, 400, 600, 800 °C; C (pin) = Superalloy IC10	• CoF and wear rate reduced with addition of Ag and $SrCO_3$ due to the formation of NiO, WO_2, and Sr_2CrO_4 at high temperature and their synergistic effect with Ag, $NiCr_2O_4$, Al_2O_3, and Cr_2O_3 in the composites.
Shan et al.[79] Tribol Lett (2024)	Hot Press Sintering Ni18Cr9W-10 wt.% Al_2O_3- (0, 5, 0 wt.%) Ag- (0, 0, 10 wt.%) $SrCO_3$	Pin on disc (Rotary) L= 10 N; V = 0.5 m/s; t = 120 min; T= RT, 200, 400, 600, 800 °C; C(pin) = Superalloy IC10	• Composite having $SrCO_3$ exhibited the improved friction and wear performance from 400–800 °C due to the formation of lubricating oxides WO_2, WO_3, NiO, and Sr_2CrO_4 and the formation of a smooth, dense and continuous self-lubricating glaze layer on the surface.
Ma et al.[80] Ceram. Int. (2024)	Hot Press Sintering Ni18Cr9W- (0.0, 1.0, 2.5, 5.0 wt.%) nano-Al_2O_3	Ball on disc (Rotary) L= 10 N; V = 0.104 m/s; t = 60 min; T= RT, 400, and 800 °C; C(ball) = Al_2O_3	• Addition of nanosized Al_2O_3 increased the CoF and wear rate of the composites at RT. However, at 400 and 800 °C, formation of Al_2O_3, NiO, and Cr_2O_3 reduced the CoF and wear rate. • The composite having 5 wt.% Al_2O_3 exhibited best high temperature tribological properties at 800 °C, with a CoF of ~0.23 and a wear rate of ~2.35×10^{-6} mm^3/Nm, (41 % as compared to the base material).

2.9 FORMULATION OF THE PROBLEM

Despite continued developments in the area of high temperature self-lubricating composites, researchers are still exploring the novel solid lubricants for realizing low friction and anti-wear properties over a wide range of temperatures for the machine elements that work under relative sliding motion. Some solid lubricants provide low friction and low wear from room temperature (RT) to moderate temperatures (≤ 500 ºC) whereas some other are effective only at elevated temperatures i.e., beyond 500 ºC. However, the requirement for the materials for being used in advanced technological systems is not only to attain low friction and low wear properties at RT but also to maintain the same at elevated temperatures (800 or 1000 ºC) and varying environments. Hence, it becomes imperative to utilize a combination of solid lubricants as reinforcements in matrix material to achieve anti-friction and anti-wear properties by optimising their content and exploring the occurrence of any synergistic action between them.

An exhaustive examination of the literature indicates that a limited studies have been conducted to evaluate the friction and wear of Ni alloy based composites containing rGO, Ni-doped rGO (rGO-Ni) and Ni-doped *h*-BN (*h*-BN-Ni) as solid lubricants or their combination with Silver (Ag) i.e., Ag-rGO, Ag-(rGO-Ni), and Ag-(*h*-BN-Ni) from room temperature (RT) to 800 °C. Silver offers lubrication up to 500 °C by providing a low shearing strength film at the sliding interface at relatively lower temperatures and by diffusing to the interface at higher temperatures. A few studies have been conducted to examine the potential of Reduced graphene oxide (rGO) as a solid lubricating reinforcement in composites. However, the homogeneous dispersion of rGO in metal matrices pauses a problem which may be overcome by modifying its surface by a metal which is a compatible with the matrix material. Also, all these studies have been carried out at room temperature only, and apparently, no study is available in the published literature that unravels the high temperature lubrication capability of rGO as a solid lubricant in Ni based composites. Hence, there is a need to explore its lubrication potential at high temperatures. Hexagonal boron nitride (*h*-BN), possessing a layered lattice structure has been reported to be a clean and high temperature lubricant due to its high thermal conductivity, excellent chemical & thermal stability, and chemical inertness at elevated temperatures. However, its poor sintering and wetting characteristics with several metal matrices have restricted its use in self-lubricating composites. The modification of *h*-BN powder with Cu or Ni has been indicated to be beneficial in improving these features.

The present study is aimed at fabricating the self-lubricating Ni alloy-based composites containing lubricating species i.e., Ag and a combination of fixed amount of Ag and different amount rGO-Ni and *h*-BN-Ni via spark plasma pressing and exploring their lubrication potential under dry sliding conditions at different temperatures i.e., RT, 200, 400, 600, and 800 °C. The rGO and *h*-BN powders have been doped with Ni individually to overcome their poor integrating properties with the matrix. The combinations of Ni with rGO and *h*-BN result in the formation of two novel nano-hybrid nanomaterials (i.e., rGO-Ni and *h*-BN-Ni), which are expected to provide enhanced lubricating capability in comparison to the unmodified ones. Both Ag and rGO-Ni are expected to provide lubrication up to moderate temperatures (≤ 500 ºC), while *h*-BN-Ni is supposed to provide low friction at elevated temperatures. The investigation also intends to examine the possibility of a synergistic relationship between Ag-(rGO-Ni), Ag-(*h*-BN-Ni), and Ag-

(rGO-Ni)-(*h*-BN-Ni) in attaining low friction and wear over an extended range temperature.

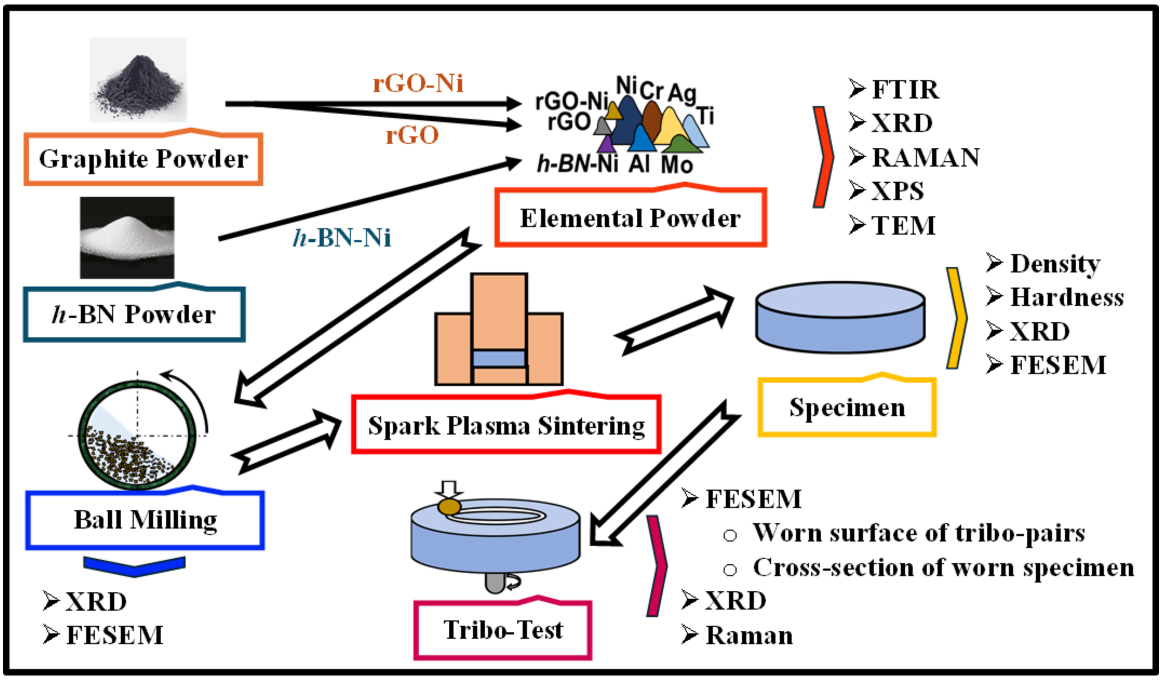

Fig. 2. 7 Schematic of step-by-step methodology of the present investigation

The current study has been conducted with the following objectives:

1. To synthesize the reduced graphene oxide (rGO), Ni doped rGO (rGO-Ni), and Ni doped *h*-BN (*h*-BN-Ni) and to characterise its structural features.

2. To fabricate Ni alloy-based composites containing fixed amount of Ag and rGO, and different amounts of rGO-Ni and *h*-BN-Ni using spark plasma sintering and to determine their content for achieving optimum tribological performance.

3. To evaluate the tribological behaviour of Ni alloy-based self-lubricating composites at RT, 200, 400, 600, and 800 °C by carrying out dry sliding wear tests at a fixed sliding load, sliding speed, and sliding time.

4. To examine the possibility of synergistic action between a combination of solid lubricants like Ag-rGO, Ag-(rGO-Ni), and Ag-(*h*-BN-Ni) in accomplishing effective lubrication from RT to 800 °C.

5. To establish the operative mechanisms of wear.