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Effect of hexagonal boron nitride and graphite on mechanical and scuffing resistance of self lubricating iron based composite



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ABSTRACT

The production of self-lubricating composites containing second phase particles is one of the most promising choices for controlling friction and wear in energy efficient modern systems. To gain a better understanding of the wear behaviour of such materials, Fe-Si-C matrix composites containing solid lubricants added during the mixing step were studied in this work. The samples were produced using powder metallurgy route, with total contents of 5, 7.5 and 10% in terms of volume of hexagonal boron nitride (hBN) and graphite mixtures as the solid lubricants. The composite's tribological properties were evaluated under reciprocating sliding conditions and their mechanical properties were tested using tensile tests. Additionally, after interruptions at different stages of the reciprocating tests, the wear scars were characterised by Raman spectroscopy and scanning electronic microscopy, to evaluate the evolution of the wear with test time. Higher total solid lubricant contents greatly increased the scuffing resistance of the composites, but decreased the mechanical properties. Furthermore, increasing the hBN content reduced both properties. Among the composites studied, the samples containing 1%vol hBN and 9%vol graphite showed the best combination of mechanical and tribological properties.

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1. Introduction

The evolution of advanced technology, social growth and concerns about the environment are driving the search for quality, economy and efficiency in new products. The development of advanced materials leads to improvements in material properties associated with high efficiency, low energy consumption, low mass, high strength, improved performance, longer product life, etc. [1]. In this context, the development of low friction, high wear resistance tribomaterials is of the utmost importance and addresses large energy losses caused by the friction that occurs in virtually every mechanical system [2].

In most general tribological applications, liquid lubricants or grease are used to prevent friction and wear. However, when service conditions become severe, (very high or low temperatures, vacuum, radiation, extreme contact pressures or conditions requiring very high degrees of cleanness, such as for foods and pharmaceuticals) solid lubricants may be the only choice [3].

Several inorganic materials such as molybdenum disulfide (MoS₂), graphite (C), hexagonal boron nitride (hBN) and boric acid (H₃BO₃) provide excellent lubrication [4–9]. These materials have a similar lamellar structure, which consists of a stack of hexagonal layers possessing strong covalent bonds between the atoms in each layer and Van der Waals weak interlayer interactions. These structures easily shear when a force is applied parallel to the layers, providing a reduction in friction [10]. A few others (e.g., soft metals, polytetrafluoroethylene, polyimide, certain oxides and rare-earth fluorides, diamond and diamond-like carbons (DLC), fullerenes) can also provide lubrication, despite not having a layered crystal structure [8–11]. A combination of solid and liquid lubrication is also feasible and may have beneficial synergistic effects on the friction and wear performance of sliding surfaces, particularly in the elasto-hydrodynamic lubrication regime [12].

The production of self-lubricating composites containing second phase particles is one of the most promising choices for controlling friction and wear in energy efficient modern systems. Such composites have been used for several decades in household and office light equipment such as printers, electric shavers, drills, blenders and others. The most commonly used metallic matrix materials are copper, ferrous and nickel alloys. The majority of the

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composites contain a high percentage of solid lubricant particles (15–40%_{vol}) to obtain a low friction coefficient. Compounds such as MoS₂, WS₂, MoSe₂, NbS₂, TaSe₂, MoTe₂ and hBN and low melting temperature metals such as Ag, Sn and Pb, graphite and polytetrafluoroethylene (PTFE) are the most often used solid lubricants [13–17]. These composite materials can be tailored using powder metallurgy [18], a processing route widely used for producing metallic parts mainly due to its low cost when used in high volume production, versatility in shape complexity and near-net-shape dimensional control [19,20].

Recently, results were presented from an ongoing research program that aims to develop new, low cost, self-lubricating materials possessing a low friction coefficient combined with high mechanical strength and wear resistance [21-23]. The mechanical and tribological performance of such sintered composites is directly related to the composite final microstructure, or, the combination of the matrix mechanical properties and structural parameters, such as the degree of continuity of the metallic matrix beyond the nature, amount, size, shape and distribution of the solid lubricant particles. To overcome the deleterious effect of matrix discontinuities in mechanical properties due to dispersed solid lubricants, the microstructure can be tailored using the versatility of powder metallurgy technique in order to obtain optimized solid lubricant reservoirs, as the mean free path among them, by selecting suitable raw materials and processing parameters [24–32].

Recently, Hammes et al. [23] reported on the effect of a double pressing/double sintering (DPDS) technique on the scuffing resistance and sliding wear of self-lubricating uniaxial die pressed hBN (5% $_{vol}$) and graphite (2.5% $_{vol}$) Fe-Si-C-Mo composites. The purpose of using both solid lubricants was associated with the tribological behaviour of the lubricants in different environments. Graphite is very well known for its lubricating effect in wet atmospheres, whereas hBN is suitable for high-temperature applications [33,34]. When two or more solid lubricants are incorporated, a synergetic lubricating effect can occur, sometimes even superior to any one of the single lubricants. Furthermore, the combination of lamellar solids with opposite solubility into the matrix, in addition with processing parameters (i.e. sintering temperature), is a potential solution to tailor the final composite microstructure. This work presents the influence of the simultaneous addition of hBN and graphite mixtures as solid lubricants on the tribological behaviour of self-lubricating composites based on a Fe-Si-C matrix produced by powder metallurgy.

2. Material and methods

In order to produce self-lubricating composites with suitable mechanical resistance and adequate lubricity, the conventional route of powder metallurgy was used. Mixtures were produced using iron (Höganäs, AHC 100.29, d50=100 μ m) as main constituent of matrix composite, silicon (Osprey Sandvik, alloy Fe45Si, d50=10 μ m) as an alloying element for stabilization of iron alpha phase and hardener of the matrix, graphite (Nacional de Grafite, Micrograph 99545HP, d50=32 μ m) and hexagonal boron nitride (Momentive, AC6028, d50=125 μ m) as solid lubricants. Table 1 detail the composites initial composition, the following parameters were varied: total content of solid lubricant in volume (5, 7.5 and 10%vol), h-BN amount (1, 1.75 and 2.5%vol) and graphite content (balance).

The powders were homogenised with 0.8%_{wt} of amide wax in a Y-type mixer (35 RPM) during 45 min, before being uniaxially pressed at 600 MPa inside a floating die and using a double-action press, to produce samples for tensile characterisation (MPIF35 [35]), cylinders for microstructural analyses (10mm diameter/

Table 1Chemical composition of the composites.

	Composite	osite Solid lubricant content (%vol)		ntent (% _{vol})	Si (%wt)	Fe (% _{wt})
_		Total	hBN	Graphite		
-	1h4C 1.75h3.25 C 2.5h2.5 C	5	1 1.75 2.5	4 3.25 2.5	0.5	Balance
	1h6.5 C 1.75h5.75 C 2.5h5C	7.5	1 1.75 2.5	6.5 5.75 5		
	1h9C 1.75h8.25 C 2.5h7.5 C	10	1 1.75 2.5	9 8.25 7.5		

6mm thickness) and tribological characterization (20mm diameter/5mm thickness). After compaction, specimens were sintered in a hybrid plasma reactor that has been developed in-house [36,37]. A debinding step was conducted at 500 °C for 30 min and isothermal sintering was carried out at 1120 °C for 1 h, both in an argon/hydrogen (95%/5%) abnormal glow discharge plasma atmosphere and using a heating rate of 10 °C/min. The microstructural analyses were performed via optical microscopy (Olympus BX60) and SEM-EDX (JSM-6390LV, Jeol — 6733a, ThermoScientific) analysis, after conventional metallographic preparation of the sintered samples.

Tensile tests were carried out according to MPIF standard 10 [35] using a MTS 810 machine and the tribological behaviour was evaluated in a CETR UMT-3 tribometer. To determine the scuffing resistance, reciprocating sliding tests were carried out using an incremental loading mode (increasing the normal load in increments of 7 N at 10 min intervals) according to the method proposed by De Mello and Binder [38]. In this study, the scuffing resistance was defined as the work (N.m) at which the value of the friction coefficient first rose above 0.2 (lubricity effect). A hard steel AISI 52100 ball (diameter 5mm) was fixed on a pivoting arm and rested against the specimen surface for reciprocating sliding, using constant stroke (10mm) and frequency (2 Hz), while the frictional force was continuously logged (acquisition rate of 1 Hz). The tests were unlubricated and conducted at a controlled relative humidity (50%) and temperature (22 \pm 1 °C). Scuffing resistance averages were calculated using data from at least seven experiments for each analysed condition. After tribological sliding evaluations, samples were ultrasonically cleaned in acetone for 15 min and the worn surfaces were investigated by SEM (ISM-6390LV, Jeol) using secondary and backscattered electrons, EDX microanalysis (6733a, ThermoScientific) and micro-Raman spectroscopy (Renishaw InVia - argon laser, $\lambda = 514.5$ nm), in order to identify the wear mechanisms in each tribological condition.

3. Results and discussion

Fig. 1 shows the typical non-etched microstructures of produced composites, with different total solid lubricant contents (5, 7.5 and $10\%_{\rm vol}$) and same amount of hBN ($1\%_{\rm vol}$). The other composites presented similar microstructural evolution with solid lubricant content. The light and continuous region is the metallic matrix while the dark one represent matrix discontinuities: pores inherent from the manufacturing technique and solid lubricant particles. Due to limitations of the technique used for image analysis it is difficult to clearly distinguish pores from solid lubricant particles and the dark regions will be called solid lubricant

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