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Thermal Stability of the MoS₂ Phase in Injection Moulded 17-4 PH Stainless Steel

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In the present paper, an analysis of the stability of the MoS₂ compound in 17-4 PH stainless steel matrix during the sintering of powder injection moulded samples is presented. A feedstock containing 10% vol. of the solid lubricant phase MoS₂, mixed with 17-4 PH stainless steel powder was prepared and injected. The sintering was carried out at various temperatures ranging from 650°C to 1,300°C. The progress of the dissociation process of MoS₂ as a function of sintering temperature and the formation of new phases were analyzed via X-ray diffraction. The microstructure of the resulting material was analyzed by SEM/EDS. As expected, for temperatures above 650°C, the results confirm the decomposition of MoS₂ and formation of others sulphides during the sintering cycle. In addition, there occurs dissolution of molybdenum resulting from MoS₂ decomposition.

KEY WORDS: Phase stability; Molybdenum disulfide; Powder Injection Moulding; Self-lubricant materials.

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1. Introduction

A solid lubricant can be applied to the components of a tribological pair in the form of films, deposited or generated in layer form on the surface, or incorporated into the volume of the matrix as dispersed particles, producing a dry self lubricating composite material^[1]. Although there are many examples of technology developed with solid lubricants as films, that kind of processing has a high production cost. Further, the use of solid lubricants in the form of films often leads to durability problems as a result of their thickness which provides only a limited reserve of lubricant. The lubri-

cation effect may cease abruptly by removing the lubricant film as a consequence of the action of mechanical stresses, especially in cases of poor adhesiveness or even as a result of wear. Considering these problems, self lubricating materials may be more appropriate for solving lubrication needs in many practical applications, where an external lubricant supply is impossible or not recommended. Self-lubricating materials are used in applications like bearings, bushings, plates, and other wear surfaces. Two distinct groups of self lubricating materials are in use: porous materials whose pores are filled with lubricant fluid and composite materials in which solid lubricant particles are dispersed in the bulk of the materials. While porous materials impregnated with fluid lubricants are the most used materials for self-lubricating bearings, the dry self-lubricating materials (solid

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lubrication) are suitable to oscillatory and linear motion as well as for high temperature and difficult working environments. Furthermore, the particular operational conditions (that are defined by the application) including the environment in which the component will be installed have to be considered in the selection of an adequate self-lubricating material. In most tribological applications it is common to use materials such as oil or greases as lubricants. However, when the operation conditions become extreme (for example: excessively high or low temperatures, vacuum, radiation, higher contact pressure etc.), these materials may not be able to satisfactorily lubricate the surfaces; in this case, the use of solid lubricants, such as molybdenum disulfide, is justified and pointed out^[2-7].

In many cases porous self-lubricating materials are inadequate because of their poor mechanical resistance, which is drastically dropped as a consequence of the high percentage of pores needed for lubricant storage, which ranges from 9% to 20% in volume of open pores. In a similar way, the strength of dry self-lubricating materials depends on the percentage of solid lubricant particles dispersed in their volume. High content of solid lubricant leads to a high degree of discontinuity in the metallic matrix, resulting in reduced mechanical strength of the composite. Thus, the correct way is to design the self-lubricating material (solid lubricant phase and content, metallic matrix and their hardness and strength, microstructure) for each specific application taking into account advanced mechanical tribological knowledge^[8]. Self-lubricating materials are normally powder materials and their production occurs via powder technology forming techniques and sintering. In the porous wet self lubricating materials, pore size and content (% vol.) are controlled by powder particle size and processing parameters while in dry self-lubricating materials the dispersion of the solid lubricant particles into the volume of the material is achieved by mixing the solid lubricant particles with the matrix powders during the feedstock preparation. An alternative way is to mix a precursor phase that leads to the *in situ* formation of the solid lubricant particles in the volume of the material. High strength combined with a low coefficient of friction was achieved in sintered steels alloyed with Si + Ni + C, using low contents of silicon carbide as a precursor for the generation of graphite through SiC dissociation during sintering^[8,9]. Traditionally, copper-based alloys (mainly bronzes) and ferrous materials (mainly plain iron and Fe-Cu alloys) are selected as a metallic matrix for both groups: wet and dry self-lubricating materials. For dry self-lubricating composites a large number of distinct solid lubricant particles can be added to the metallic matrix. The most effective and used materials are graphite and lamellar compounds, such as MoS₂, WS₂, MoSe₂, and hBN, as well as low melting point metals such as Ag, Sn, and Pb^[2,7]. In this kind of composite material, the production cost is pretty much similar to that of the original part (part without solid lubricant), since there is no need of additional fabrication steps; the solid lubrication source is in the whole volume of the part. Hence there is an abundant quantity of solid lubricant and usually the geometry of the part has no influence on its performance.

The selection of the solid lubricant phase depends upon the matrix in order to avoid undesirable reactions between both materials. For example, the compound MoS₂ is widely

used in combination with a copper or bronze matrix, but its use in combination with ferrous matrices will result in reaction of the sulphur with iron, forming iron sulphide (FeS), which is more stable than MoS₂ at the high temperatures needed for the sintering^[3].

Several studies of dry self-lubricant composites manufactured by powder metallurgy with MoS₂ in a variety of matrices, such as Ni-Cr-W-Fe-C^[4], 316L^[10], AISI M3/2 and M35^[11], Ni-Cr-W-Al-Ti^[12], Fe-C-Cu^[13], nickel^[14], and bronze^[15] have been conducted recently. The results include mechanical^[4,11,13-15], microstructural^[4,10-12], and tribological characterization^[4,10,12-15].

In this work, composites of 17-4 PH (matrix) with additions of MoS₂ were prepared by powder injection moulding and sintered at various temperatures, ranging from 650 °C to 1,300 °C. The aim of this procedure was to analyze the progress of the dissociation of MoS₂ in the 17-4 PH steel matrix as well as the resulting new phases formed as a function of sintering temperature. The alloy 17-4 PH is a precipitation hardenable martensitic stainless steel, which is recognized for its high strength and corrosion resistance and has widespread applications, such as aeronautic, automotive, marine, military, and medical instruments areas^[16-19]. The precipitation hardening treatment provides high hardness to the alloy, resulting in difficulties during the machining step in the conventional process. As a way to overcome that, the powder injection moulding is an excellent alternative for the manufacture of 17-4 PH stainless steel components^[16,20,21].

2. Materials and Methods

Water atomized metallic powder of 17-4 PH (Atmix Co. Ltda, d₅₀ = 10 μm) and MoS₂ powder (Jet Lube, d₅₀ = 12 μm) were mixed, in a volumetric proportion of 90% 17-4 PH/10% MoS₂. The chemical composition of the 17-4 PH steel is 15.75 %wt Cr, 4.24 %wt Ni, 3.27 %wt Cu, 0.30 %wt Nb, 0.05 %wt C, 0.82 %wt Si, 0.83 %wt Mn, 0.02 %wt P, 0.01 %wt Mo, 0.05 %wt Co, Fe balance. A feedstock for powder injection moulding was prepared by adding 8 %wt of a binder system composed by polymers and waxes (sigma blade mixer, Hake Rheomix, 180 °C, 60 m, 1.17 Hz). After cooling, the feedstock was grinded in a cutting-blade mill (Seibt).

For the injection of the parts an Arburg Allrounder 320S injection machine was used. The debinding was carried out chemically (Hexane 60 °C - vapour 2 hours, immersion 5 hours) and thermally in an abnormal glow discharge hydrogen plasma (1 Torr, 400 V). After the debinding, the sintering was carried out in a tubular resistive furnace (AISI 316 stainless steel tube as sintering retort) with a 95% Ar 5% H₂ controlled atmosphere. The heating rates were 10 °C/min (up to 650 °C) and 5 °C/min, up to the sintering temperature. The isothermal sintering was carried out at different temperatures: 650 °C; 750 °C; 900 °C; 1,150 °C to 1,300 °C. The cooling down was done as fast as possible, reaching cooling rates over 25 °C min⁻¹.

For the microstructural characterization, the samples were metallographically prepared and the microstructure revealed with Marble reagent solution. The samples were analyzed chemically and morphologically before and after etching using an optical microscope Leica MD4000M and a scanning electronic microscope (SEM) Philips XL30 with

EDAX. The X-ray diffraction (XRD) analyses were performed in a Philips X'pert equipment, with Cu K α (1.54056 Å) radiation, parameters: 40 kV and 30 mA. Three samples, without any post-sintering heating treatments, were used for each condition.

The tribological characterisation was carried out in air in an unlubricated reciprocating wear test (UMT-CETR), in which both contact potential and friction force were continually logged with a microcomputer. A hard steel sphere was held on a pivoted arm, and rested against the specimen surface under constant stroke (10 mm), frequency (2 Hz), and normal load (7 N). The tests were conducted under controlled room temperature (20 °C) and humidity (50%) conditions. The ball surface was used in the as-received condition and a new surface region was used for each test. Before each test, balls and samples were thoroughly cleaned first with ethanol and then by acetone in an ultrasonic cleaner, after that they were dried in hot air.

3. Results and Discussion

3.1 Microstructure and EDAX Analysis

The 17-4 PH samples without solid lubricant (Fig. 1) presented a martensitic microstructure. The sample sintered at 1300 °C presented pores with rounded shape, in comparison to the one sintered at 1,150 °C.

The samples with solid lubricant (Fig. 2) presented diverse microstructures. The sample sintered at 1,150 °C shows probably a ferritic microstructure with second phase particles homogeneously dispersed into the matrix. The sample sintered at 1300 °C, besides presenting a martensitic matrix, also shows grain growth as well as second phase coalescence and, as a consequence, larger size in comparison with the sample sintered at 1,150 °C.

For the samples sintered at 1150 °C (Fig. 2a), a martensitic microstructure was expected; however, this was not found in our analysis. This could have occurred due to

low cooling rate, which in this case would be lower than that needed for martensite formation. In addition, Mo released in the decomposition of molybdenum disulfide is dissolved in the matrix and, consequently, increases the tendency to stabilize the ferritic structure. In the samples sintered at 1,300 °C (Fig. 2b), the typical martensitic microstructure of 17-4 PH steel was found. The sample sintered at 1,300 °C presented also a diverse microstructure in the bottom of the sample, a layer with a weak bonding to the sample nucleus (Fig. 3). It contains copper in a great extent (according to EDAX analysis) and ~160 µm in thickness.

Due to its low mechanical strength after sintering, it was not possible to prepare the samples sintered at 650 °C, 700 °C, and 900 °C for microstructural analysis.

The EDAX analysis of the samples sintered at 1,150 °C shows that part of the molybdenum released from MoS₂ decomposition has already been dissolved by the matrix at this sintering temperature. Indeed, the chemical analysis indicated 3.3 %wt Mo (before sintering the Mo content of the 17-4 PH steel is <0.01 %wt). The remaining of Mo is found in the white particles ('A', Fig. 4) which are molybdenum rich (~34 %wt), indicating that the MoS₂ could have dis-

Fig. 1 Micrograph of 17-4 PH samples etched with Marble. Sintered at (a) 1150 °C; (b) 1,300 °C

Fig. 2 Micrograph of 17-4 PH + 10%vol MoS₂ samples etched with Marble. Sintered at (a) 1,150 °C; and (b) 1,300 °C

Fig. 3 Micrograph of 17-4 PH + 10%vol MoS₂ samples sintered at 1,300 °C. Bottom segment of the sample

Fig. 4 SEM-BSE of 17-4 PH + 10% vol. MoS₂. Core etched with Marble. Sintered at 1,150 °C

sociated and the sulphur have formed another compounds. Ni, Cr, and Fe were also found in some of these particles, the elements, which indicate that Mo may have formed another phases too. The visible phase in the grain boundaries has a composition similar to the matrix. In this sample there are three types of particles spread in the matrix, indicated in Fig. 4 as B, C, and D. The particles 'B' have a composition similar to the matrix, but with a higher level of sulphur (2.6 %wt versus 0,0%wt in the matrix). The 'C' particles (tiny size, rounded) have high content of Mo (10 %wt), Cr (40 %wt), and S (14 %wt), denoting the possible formation of chromium sulphide and/or a complex chromium-molybdenum sulphide, besides part of the sulphur could still be linked to the Mo. The particles 'D' found on pore edges also present high content of Mo (25 %wt) and Cr (22 %wt), however low sulphur levels (1,7 %wt).

3.2 XRD Analysis

The XRD analyses were carried out on the powder mixture and on the sintered samples. For all the studied temperatures, it aims to verify, comparatively to the 17-4 PH steel without MoS₂, the phases formed and their evolution with the increase of the sintering temperature, as well as the formation or dissociation of phases. The XRD of the samples sintered in 1,150 °C and 1,300 °C were analyzed in detail. The powder XRD pattern (Fig. 5) presents three different phases: ferrite (α), austenite (γ), and molybdenum disulfide. The three phases were expected due to the physical mixture between the MoS₂ and 17-4 PH powders.

The evolution of phases according to the sintering temperature is visualized in Fig. 6. In this graph, the phase indicated as 'A' could be assigned both to the ferrite and martensite structure, since these two phases have a similar crystalline structure leading to overlay of XRD patterns^[22].

At 650 °C, the presence of the molybdenum disulfide characteristic peak is observed closely to $2\theta = 14^\circ$, as well as the other peaks (indicated by the letter C). With an increase of 100 °C in the sintering temperature, the characteristic peak disappears, indicating the decomposition of MoS₂ and

Fig. 6 XRD patterns of 17-4 PH + 10% vol. MoS₂ - evolution of phases according to the sintering temperatures

the possible formation of other sulphides, whose peaks are indicated by a circle in Fig. 6. The samples sintered at 900 °C exhibit similar behaviour to the ones sintered at 750 °C. The diffractogram of the samples sintered at 1,150 °C presents four of the six peaks attributed to the austenitic phase (B) and an intense peak in $2\theta \approx 44^\circ$ together with other three peaks attributed to the ferritic phase (A). These results are verified by the metallographic analysis of the sample that shows no formation of martensite and possible retained austenite. In this diffractogram, other peaks of lower intensity can also be observed. These peaks are associated with sulphides, detailed afterwards. At 1,300 °C the samples present the peaks of the martensitic phase (A) and other small peaks identified as sulphides. These peaks differ from that found at lower temperatures, which suggests that temperature increasing causes the formation of new sulphide phases which differ in composition or stoichiometry from those found before the reaction happens. It is also possible to observe that those sulphides peaks that have occurred in XRD pattern of samples with MoS₂ are not present in the samples of the steel 17-4 PH sintered without lubricant (light grey patterns in Fig. 6). These peaks are sulphides, but not molybdenum sulphide, once the most intense peak of this phase it is not shown at any temperature above 650 °C, suggesting that the MoS₂ has decomposed, forming other sulphides.

The detailed XRD pattern of the samples sintered at 1,150 °C and 1,300 °C are presented in Figs. 7 and 8.

The sample sintered at 1,150 °C does not presents the characteristic peak of molybdenum disulfide ($2\theta \approx 14^\circ$), what indicate that this phase has disappeared. The austenitic phase (A) is easily identified, by presenting four of six peaks referring to this phase. The ferritic phase (B) is identified by the presence of the most intense peak ($2\theta \approx 44^\circ$) and other three peaks associated to this phase. The remaining phases are difficult to identify as the most intense peak is overlaid to the ferrite peak and less intense peaks occur for the same angles for several phases. The chromium sulphide phase (C) has its most intense peak ($2\theta \approx 43^\circ$) overlaid by the austenitic phase peak (A). Moreover other peaks of this phase are coincident with the iron sulphide phase

Fig. 5 DRX of 17-4 PH and MoS₂ powder mixture

Fig. 7 XRD pattern of 17-4 PH + 10% vol. MoS₂ sintered at 1,150°C

Fig. 8 XRD pattern of 17-4 PH + 10% vol. MoS₂ sintered at 1,300°C

(D). Through this isolate analysis it is impossible to be sure about the presence or absence of this phase. Even so, it is possible that this phase is present including or excluding the other phase (D), i.e., it is possible to have only one of them or both phases. The iron sulphide phase has its most intense peak also overlaid by the austenitic phase peak (in $2\theta \approx 43^\circ$). The iron molybdenum phase (E) presents its most intense peak in $2\theta \approx 22^\circ$; however, it is possible to identify only one other peak of this phase. Nevertheless, by the chemical composition restrictions of the elements that should exist in the sample (17-4 PH SS plus MoS₂) this is the only phase that fits the peak found in $2\theta \approx 22^\circ$.

The sample sintered at 1,300°C also does not present the molybdenum disulphide characteristic peak ($2\theta \approx 14^\circ$). The martensitic phase (A) is readily identified, with the presence of the most intense peak and other three peaks referring to this phase. The chromium sulphide phase (B) has its most intense peak ($2\theta \approx 43^\circ$) concurrent with the iron sulphide (D) phase. The other peaks are also similar, thus,

as in the sample sintered in 1,150°C, through this isolate analysis it is not possible to be sure of the presence or absence of this phase or the iron sulphide phase. The chromium-nickel sulphide phase (C) has its most intense peak in $2\theta \approx 34^\circ$; however, this peak is a coincident peak with both B and D phases and some of the other peaks are also coincident; therefore, the sample could contain only one phase, B or C or D, or all of them.

3.3 Tribological Analysis

The tribological analysis was carried out in the samples sintered at 1,150°C with and without MoS₂ addition.

The results (Fig. 9) had shown that there is no difference in the tribological behaviour, referring to the friction coefficient, between the two samples. The addition of MoS₂ had not improved the lubricant properties of the part. This may have occurred because MoS₂ had decomposed and the new sulphides formed do not hold good tribological characteristics, which in this case would mean, low friction coefficient.

4. Conclusions

The results of XRD analysis of the sintered samples show XRD patterns composed by a variety of peaks, most of them coincident for the diverse phases. The samples sintered at 900°C, 1,150°C, and 1,300°C present the ferrite and/or austenite phase with peaks displacement, indicating a structure distortion, owed to, probably, a solid solution of some element into the matrix^[22]. They may also contain Ni, Cr and Fe sulphides and for the sample sintered at 1,300°C, complex sulphides, in agreement with previous results reported in other studies^[4,11,12,15,23,24]. The sample sintered at 1,150°C also presents a FeMo phase in the XRD pattern. The EDAX analysis showed that molybdenum has diffused into the iron matrix, so, it is very likely that this phase is present. Combining the results of EDAX and XRD, it is believed that, in the sample sintered at 1,150°C, occurs the

Fig. 9 Friction behaviour of 17-4 PH + 10% vol. MoS₂ sintered at 1,150°C during the reciprocating wear test

Fig. 10 Ellingham diagram of sulphides. Adapted from ref: Adamian, et al.^[25] and Bradley et al.^[26]

formation of chromium sulphide, with decomposition of molybdenum disulfide, or further the formation of iron sulphides. For the sample sintered at 1,300 °C it is believed that also occurs the formation of chromium sulphide or iron sulphide. The disappearance of the FeMo phase in the XRD spectra could be attributed to the raising of temperature inducing the complete diffusion of Mo in the iron matrix.

Analyzing the Ellingham diagram for sulphides (Fig. 10), it can be verified that, at the processing temperatures, the Cr sulphide has higher stability than the Fe sulphide, what suggests the preferential formation of the first one. Still in the diagram, it can be seen that for temperatures higher than 1,100 °C the chromium and iron sulphide becomes more stable than the molybdenum disulfide, with the decomposition of MoS₂ and formation of the other sulphides. Previous studies confirm that^[24]. For iron alloys with MoS₂ addition, the temperature which is attributed the formation of iron sulphide is 700 °C-900 °C, the earliest forming a non-stoichiometric iron sulphide (Fe₃S₄) which later transforms into FeS^[3,24].

To summarise:

- The samples of 17-4 PH with 10% in volume of lubricant MoS₂ have presented diverse microstructures and phases according to the distinct sintering temperature employed;
- according to the XRD analysis result, MoS₂ remain stable in 17-4 PH steel matrix up to sintering temperatures of 650 °C.
- in the case of samples sintered at 750 °C, 900 °C, 1,500 °C, and 1,300 °C, MoS₂ decomposes during sintering and Mo is gradually dissolved in the 17-4 PH matrix, while sulphur reacts with iron as well as with alloying elements present in the steel matrix, forming sulphides, mostly Cr and Fe sulphides.

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