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Influence of residual porosity on the dry and lubricated sliding wear of a powder metallurgy austenitic stainless steel

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ABSTRACT

Powder metallurgy practices offer the opportunity to control the porosity of steels for tribology applications. The material of interest was an austenitic powder metallurgy stainless steel sintered in a nitrogen atmosphere. Four disc samples with increasing porosity (from 8% to 29%) were subjected to dry and lubricated sliding against an alumina pin using a rotary pin-on-disc tribometer. An estimation based on Hertzian contact analysis has been introduced to define the size of large pores. A normalized scar depth has been defined. The influence of pores on Archard's wear law has been analysed. Dry sliding, liquid lubrication, and self-lubrication (filled pores) were all evaluated. During the first few metres of dry sliding, the pores are closed by plastic deformation. After that, the wear rate can be considered the same. In lubricated conditions, pores are not closed and the higher the porosity, the lower the wear rate. In filled-pore, self-lubricated conditions, the sample with the highest porosity showed the lowest wear rate. Reasons for this behaviour are discussed.

1. Introduction

Porous austenitic stainless steel (SS) can currently be obtained by powder metallurgy (PM) techniques. Powder metallurgy processing provides a feasible and economic way of manufacturing austenitic stainless steel (SS) components with a complex shape and such advantages as good dimensional precision, high surface finish and acceptable mechanical properties. A progressive increase in austenitic PM SS components has also been observed in recent years in such industries as aerospace, agriculture, building, construction, chemical industry, biomaterials, etc. [1–4].

Porosity can be controlled by controlling the powder size and compaction and sintering conditions. Porosity can be positive but also presents drawbacks. First, it reduces yield strength and other mechanical properties; second, it decreases the corrosion resistance; and third, it creates surface defects that can wear and develop into fatigue cracks [5,6]. The influence of porosity on wear behaviour is still a matter of discussion. Pores decrease the real contact area between two sliding surfaces. Consequently, stress distribution and metal deformation of subsurface layers are affected [7–9]. At large porosity, pores are interconnected, the strength is lower, and these pores are much easier to deform, [10] provided of course that the material is ductile. It is believed that pore size is more critical than the overall porosity content [11,12]. In addition, pores are lubricant reservoirs under wet sliding conditions, which may be noted as a major advantage in processes of wearing [13]. Some authors [14,15] have found that, in dry sliding conditions, the wear rate increases along with the increasing volume fraction of the porosity. The trapping of wear debris in the pores decreases wear rate by decreasing the contact pressure [16–18]. For high porosity levels and large pore sizes, plastic deformation results in filling the pores with wear debris [17,19,20] and, after all, they can be closed by plastic deformation.

In terms of the chemical composition of SS, it has been reported that the nitrogen addition to austenitic SS increases wear resistance [21–23]. In this study, a nitrogen–hydrogen (95–5%) atmosphere was used during the sintering process. Previous works have proven that austenitic SS sintered in nitrogen shows good dry wear resistance [24]. It has also been shown that nitrogen is an important alloying element in terms of the improvement of corrosion resistance and strength. It promotes passivity and widens the passive range [25,26]. Salahinejad et al. [27] have studied the dry sliding wear characteristics of porous austenitic Cr–Mn high-nitrogen nickel-free SS obtained by mechanical alloying and compared them with non-porous 316 SS. They concluded that the dry wear weight loss was similar for both materials and that delamination was the main wear mechanism observed on porous samples. High-nitrogen SS showed higher wear resistance than conventional SS for different media such as water, Hank's solution or NaCl solution.

In this work, austenitic PM SS with four different porosity values are investigated. These are chosen to determine the influence of porosity on wear behaviour. The main issue is to evaluate the effect

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of large pores due to the fact that they can be more positive for wear behaviour. In this way, three of the four samples present porosity in the range between 22% and 29%. Furthermore, dry sliding condition is the main scope of the work. However, lubrication is also a matter of consideration. Therefore, as a second part of this contribution, a carboxymethyl cellulose fluid of similar rheological properties to synovial fluid [28] is chosen. A biological lubricant is chosen based on the interest of the research group for further works, which will be focused on the study of porous materials for biological applications.

2. Materials and methods

2.1. Materials and characterization

Three stainless steels with slight differences in chemical composition, but with different particle sizes, have been used, Table 1. They were prealloyed and water atomized AISI 316L SS powders were supplied by HÖGANÄS, Belgium. The first, designated as SP (small particle size), was the standard AISI 316L SS with a nominal particle size lower than 150 μm. The second material, named MP (medium particle size), was a 316L SS with particle size between 300 and 500 μm. The last one was 316LF and this was designated herein as LP (large particle size); its nominal particle size was 500–710 μm. Furthermore, a fourth type of sample was obtained by mixing 50 wt% of MP and 50 wt% of LP, designated herein as MLP.

Disc specimens (12 mm diameter and 6 mm thickness) were uniaxially compacted at 550 MPa using a floating die. Zinc stearate was used as die lubricant. Green compacts were sintered in N_2–H_2 (95–5%) at 1250 °C for 60 min and cooling rate of 5 °C/min. In this work, no post-sintered heat treatment was applied. The compaction pressure was set in such a way that MP, MLP, and LP would show porosity higher than 20%. After sintering the nitrogen content was around 0.35 wt% for every sample. Image analysis was used to study this porosity. Seven fields like those presented in Fig. 1 were taken per sample. Images were digitized and calibrated. The pores were identified as the black pixels and they were computed to calculate the area of each pore, Table 2 and Fig. 2. The microstructures were revealed by metallographic preparation. After each grinding or polishing step the samples were ultrasonically cleaned to ensure the pore network remained open.

The apparent hardness (i.e. the value obtained when indentation is the result of plastic deformation of the structure and the pores) was evaluated by the Vickers method, using a load of 30 kp (294 N) during 30 s. An average value of five indentations was given as the hardness value.

2.2. Wear testing

Wear tests were carried out at room temperature using a rotary pin-on-disc tribometer following the ASTM G99-05 standard test method [29] under dry sliding conditions. An alumina ball with a 6 mm diameter and Vickers hardness of 1500–1650 was used as counter-body. A new alumina ball was used for every experiment. It was found that their wear volume was very low, less than 0.5% the wear volume of the steels. This wear does not affect to the conclusion of the work but the scar depth values were slightly overestimated. The discs were the sintered materials. Prior to starting wear tests, the specimens were polished using abrasive papers up to 1 μm diamond and ultrasonically cleaned. Every test was conducted with a sliding velocity of 0.1 m/s, a sliding distance of 200 m, and a normal load of 10 N. Real time wear scar depth was measured by a capacitive displacement sensor attached at the tribometer's arm carrier.

A set of samples were tested under discontinuous lubricant conditions. A 0.5% aqueous solution of carboxymethyl cellulose sodium salt medium viscosity (CMC) was chosen as lubricant. Lubrication was achieved by means of a spray dispenser and the lubrication cycle was set to 3 s of spray and 5 s of standby.

Finally, another set of tests was done in self-lubrication conditions by filling pores with the same lubricant as for the fully-lubricated tests. The porous samples were saturated with 0.5% of CMC aqueous solution. The samples were impregnated in a vacuum chamber during 12 h to assure that pores were full of lubricant. Some other lubricant concentrations and impregnation times were tested before setting these operational parameters. This lubricant was chosen because a potential application of these porous materials (after homogenizing their microstructure) would be in biomedical implants, though this is a matter for future works.

During the tribological tests, the kinetic coefficient of friction (COF) and the scar wear depth (SD) were continuously recorded. The SD parameter was chosen as a measure of wear rate. A more conventional parameter to determine wear rate would be the loss of material per unit of sliding distance per unit of applied normal load. However, due to the intrinsic porosity of the samples, this parameter would be inaccurate. The scar wear depth is a direct experimental measurement and it may thus be considered more reliable. Finally, the morphology of the wear surface was observed under scanning electron microscopy (SEM). All pin-on-disc tests were repeated three times to ensure repeatability.

3. Results and discussion

3.1. Porosity and microstructural studies

Wear behaviour is strongly conditioned by the kind of material, its microstructure and the possible presence of pores. This last factor is mainly the one being evaluated in this work. In Fig. 1, optical micrographs of polished samples are shown. As expected from the experimental setup, SP showed the lowest porosity, while the mixture of the two large powders (i.e. MLP) showed an increase in porosity, Table 2. After this, MP even showed higher porosity, and finally, LP was the one with the highest porosity. Consequently, the measured porosity correlates with the initial particle sizes of the powders. Therefore, in this study, four PM samples with increasing porosity are managed. Among them, MLP, MP, and LP clearly show a picture of large pores. It should be pointed out that the SP sample is mainly taken as a reference, in terms that its porosity is clearly much lower. As a working hypothesis, large pores should facilitate circulation on lubricating fluid and, therefore, a better wear behaviour would be expected as porosity increases.

Pore size distribution is also a point of interest since, not only might the total porosity have an influence on the tribological behaviour, but also the size of the pores. Fig. 2 shows the pore size

Table 1
Chemical compositions of 316L metallic powders.

Sample	Particle size (μm)	Apparent density (g/cm^3)	C (wt%)	Si (wt%)	Mn (wt%)	Cr (wt%)	Ni (wt%)	Mo (wt%)	Others (wt%)	Fe
SP	< 150	3.10	0.021	0.87	0.20	17.1	13.55	2.24	0.02 (Cu)	Bal.
MP	300–500	1.86	0.026	0.90	0.10	17.6	12.60	2.20	0.009 (S)	Bal.
LP	500–750	1.70	0.020	0.90	0.10	17.4	12.20	2.10	0.010 (S)	Bal.