ON THE USE OF FRACTAL GEOMETRY CONCEPTS FOR EVALUATING THE WEAR BEHAVIOUR OF DUCTILE MATERIALS. APPLICATION TO Mn Mo SINTERED STEELS.

M. Kupková, M. Actis Grande, M. Kupka, E. Dudrová, M. Rosso

Abstract

The evolution of friction and wear of Fe-2Mn-0.85Mo-0.5C steels sintered under two different conditions was investigated during dry sliding tests. Irregular behaviour of the friction coefficient was observed and analysed by means of fractal geometry methods. It was proven that the friction coefficient traces had the property of fractal curves. In general, the fractal dimension of friction coefficient trace tended to increase with increasing wear rate. But as the samples investigated were capable of plastic deformation, the measured mass wear rate revealed itself as not a very proper quantity for evaluating the wear process intensity. The volume wear rate seems to be more promising candidate for an appearance in a prospective correlation between the intensity of frictional wear and fractal characteristics of the evolution of frictional force.

Keywords: wear, Mn Mo sintered steels, fractal geometry, friction coefficient traces

INTRODUCTION

Mechanical devices very often contain adjacent parts that are sliding against one another during the device is operation. As a result, a progressive damage and material loss occur on corresponding component surfaces: a wear of machine parts takes place. This represents an important problem in the operating life of devices. And as such, it has serious economic consequences. Therefore, considerable efforts have been expended on the development of deterministic models which could enable engineers and designers to predict the lifespan of a product with confidence. Unfortunately, no simple and universal treatment is applicable to all situations [1,2], and the development of a reliable predictive theory of wear process is still a scientific and industrial challenge.

The continuing deficiency of a suitable model is due to wear process complexity, which is linked to a great number of properties and variable parameters which affect wear behaviour. Nonlinear processes, sudden transitions from mild to severe wear with extreme jumps in wear rates, significant plastic deformation shown by materials usually classified as brittle, peculiar chemical interactions at interfaces - these are only some of the phenomena illustrating the non-triviality of wear process. Recognizing all this complexity, the process leading to the understanding of wear behaviour resembles the creation of a mosaic consisting of many tiles. Under these circumstances, every piece of knowledge concerning at least a partial tribological problem represents a valuable contribution.

Miriam Kupková, Eva Dudrová, Institute of Materials Research, Slovak Academy of Sciences, Košice, Slovak Republic

Marco Actis Grande, Mario Rosso, Politecnico di Torino - Sede di Alessandria, Alessandria, Italy Martin Kupka, Institute of Experimental Physics, Slovak Academy of Sciences, Košice, Slovak Republic

Recently it was proposed [3,4] that possible fractal fragmentations and damages of (sub) surface material during the sliding test can reveal themselves in fluctuations in the stress-strain state of material. This can result in intense fluctuations in surface traction force and consequently in the friction coefficient connected with this force. Due to the above matters, the dependent friction coefficient vs. sliding distance (time) can possess a fractal character and its analysis could provide information on character and intensity of the damage mechanism.

During each wear test period, the evolution of the friction coefficient is similar practically for all materials. Within a short running-in stage, the friction coefficient increases and reaches a steady-state value, which is then maintained during the entire test. However, very intense fluctuations of the friction coefficient around the mean value may be present at the steady state. And only these fluctuations are object of interest.

The possible fractal character of friction coefficient traces has already been investigated for some brittle materials. In that context, data from a tribological system consisting of a $\mathrm{Si}_3\mathrm{N}_4$ ball dry sliding against a $\mathrm{Si}_3\mathrm{N}_4/\mathrm{SiC}$ nanocomposite disc were analysed [3,4]. The analysis confirmed that the friction coefficient traces were fractal curves, and their fractal dimension increased with increasing wear rates measured in corresponding sliding tests (Fig.1).

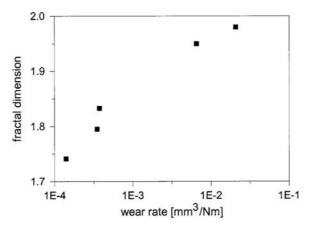


Fig.1. Fractal dimensions of friction coefficient traces vs. wear rates for a system consisting of Si₃N₄ ball dry sliding against the Si₃N₄/SiC nanocomposite disc. (Sliding velocity 0.1m/s, applied loads 10, 15, 20 N and sliding distances 600, 900 m).

In the contribution presented here, it has been investigated whether the friction coefficient evolution reveals fractal character, also for ductile samples, and what kind of information on the wear process could be extracted from the subsequent "fractal" analysis in this case. In that context, some results of dry sliding tests carried out on Mn Mo sintered steels were analysed.

The above mentioned "fractal" analysis represented in some sense a "by-product", an additional unconventional processing of data, as the experiments were primarily aimed at building up the conventional tribological data on Mn sintered steels as a part of the material and process developed. The effect of two different sintering conditions were attended to, one of them related to a new proposed type of sinter-hardening process based

on vacuum sintering coupled with integrated rapid cooling, and the second one represented by the conventional "industrial" sintering.

Sinter-hardening proves to be an attractive process for some special applications. It requires cooling a part from the sintering temperature at a rate sufficient to transform a significant portion of the material matrix to martensite. Such a process is rather attractive for components difficult to be quenched because of their shape or dimensions and some important advantages can be derived from its use. Sinter-hardening allows for the production of powder metallurgy components having high apparent hardness and high strength, thus reducing the need for secondary heat treatments [5-8]. This aspect could make PM products even more attractive, especially in the case of applications where high wear resistance is required.

In regards to the materials which have been studied, an advantage could be the possibility to substitute expensive, carcinogenic and toxic nickel and nonrecyclable copper by introducing manganese into sintered steels [9,10]. In addition, manganese improves the strength of ferrite and increases the hardenability of steels. Its application in PM products encounters, some difficulties however, mainly related to its high affinity for oxygen [11]. To prevent the formation of oxides, a proper sintering atmosphere is required during the sintering cycle. The presence of molybdenum as an alloying element is very important; in fact, it is helpful to improve hardenability and the mechanical properties of steel by solution hardening and carbide precipitation [12,13].

MATERIALS AND PROCEDURES

Preparation of samples

Within the framework of the investigation of PM Mn steels, a number of samples were prepared using different powder mixes based on Fe-Mo-Mn and Fe-Cr-Mo-Mn systems. The raw materials employed in this investigation were as follows: commercial water atomised prealloyed powder (Fe-0.85% Mo, Höganäs Astaloy85Mo), commercial medium carbon ferromanganese (Fe-77Mn-1.4C-1.3O) (from Elkem Ferromanganese Sauda, Norway) milled under a N_2 protective atmosphere to yield 56% particles under 10 μ m mean diameter, commercial CR 12 graphite powder (from GRAFITE, Netolice) with particle size less than 40 μ m, and commercial HW wax powder as lubricant.

The nominal composition of samples chosen for "fractal" analysis was Fe-2.0 wt.% Mn-0.85 wt.% Mo-0.5 wt.% C. This composition should provide proper samples [10,14]. Required powders were mixed using a laboratory turbular mixer for 20 min. Samples were obtained using a 2000 kN hydraulic press, in a disc-shaped mould (40 mm diameter), applying a pressure of 600 MPa.

Two different sintering cycles were applied:

- Sintering in an industrial Cremer pusher furnace (Metalsint a.s. Dolný Kubín) in an atmosphere of $25\%~H_2 + 75\%~N_2$, obtained from a cryogenic liquid.
- Sintering in a high temperature vacuum. In this case, a rapid cooling from sintering temperature was applied within the cycle by means of nitrogen flowing gas at 4 bars pressure, with an integrated final tempering.

Tab.1. summarises the parameters of the two different sintering processes.

| | Industrial sintering | Laboratory sintering | |
|-----------------------|---|-------------------------------|--|
| Furnace | Cremer pusher furnace | TAV | |
| Sintering time | 40 min 60 min | | |
| Sintering temperature | 1180°C | 1240°C | |
| Cooling rate | 10°C/min | 350°C/min (900°C to 300°C) | |
| Atmosphere | 25H ₂ +75N ₂ (pure gases-flowing gases) | Vacuum + argon back filling | |
| Dew point | -55°C | - | |
| Tempering | - | 180°C – 60 min | |

Tab.1. Sintering parameters.

EXPERIMENTAL PROCEDURES

Densities were evaluated using the water displacement method.

Microstructure observations were carried out using LEICA MEF4A light microscope. The apparent hardness of the test specimens (HV 10) was determined by means of a Vickers hardness indenter.

Pin-on-disc test was carried out by means of a tribometer entirely developed in the Alessandria Campus of Politecnico di Torino. The disc was made of the material investigated. As a counter face, a WC-Co pin was used, having a rounded shape on top with a 3 mm diameter. The counter-pin was changed after the end of each test, in order to preserve the roundness of its top. All wear tests were performed in air and without any lubricant. The applied loads were 5 N and 15 N. The rotation speed of the disc was 300 rpm. The distances of the pin position from the disc centre were 15 or 17 mm.

All samples were tested on both sides, after having been polished with abrasive papers in order to determine a medium surface roughness equal (or less) to $0.8~\mu m$, as specified in the ASTM G99 – 95a.

Each test was interrupted after 100, 300, 600, 1100 and 2100 meters sliding distance and discs were weighed (using precision scales with a sensitivity of 10⁻⁵ g) to determine the evolution of the wear during each test.

The evolution of friction coefficients during each test was recorded.

The total wear volume was evaluated from a careful analysis of the final wear track profile. This profile was estimated by means of light micrographs of cross section through the worn surfaces.

Data processing

Observed evolution of the friction coefficient revealed an irregular character. As its fluctuations around the mean value have been possibly raised by the fractal failure of subsurface material, the friction coefficient evolution can represent a non-stationary process. Such signals are quite difficult to process properly in a standard way (e.g. by means of Fourier analysis). But a fractal-based study can still be carried out.

A variety of algorithms are available for the computation of fractal dimension. Higuchi's estimator [15] was chosen here as it is one of the most robust methods to compute the fractal dimension of discrete time series and provides the most accurate estimates of this dimension [16]. This algorithm is briefly sketched below.

Consider X(1), X(2),...,X(N) the time sequence to be analysed. From a given time series, k new time series X_m^k are constructed, defined as follows:

$$X_{m}^{k} = \{X(m), X(m+k), X(m+2k), ..., X(m+n_{li}(N, m, k)k)\}$$
 for $m=1,2,...,k$.

Here $n_{ll}(N,m,k)$ stands for the lower integer part of (N-m)/k. m indicates the initial time value, and k represents the discrete time interval (delay) between points. For a time interval equal to k, one gets k sets of new time series. For each of the curves or time series X_m^k , the length of the curve is defined as follows:

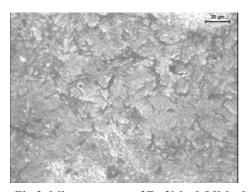
$$L_{m}(k) = \frac{N-1}{n_{li}(N,m,k)k^{2}} \sum_{i=1}^{n_{li}(N,m,k)} |X(m+ik) - X(m+(i-1)k)|.$$

The term $(N-1)/n_{li}(N,m,k)k^2$ represents the normalization factor for the curve length of the subset time series. The length < L(k) > of the curve for the time delay k is defined as the average value of k lengths $L_m(k)$. If $< L(k) > \infty k^{-D}$, then the curve analysed is fractal with the dimension D.

The fractal dimension D can be understood as a "measure of roughness" of the curve considered. The higher the fractal dimension, the more rough and irregular the curve is.

RESULTS AND DISCUSSION

Figure 2 shows the microstructures obtained by sintering the Fe-2Mn-0.85Mo-0.5C material under the two different conditions. After being polished, metallographic sections were etched with nital 3%.



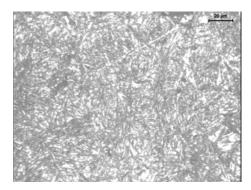


Fig.2. Microstructure of Fe-2Mn-0.85Mo-0.5C material sintered a) in N_2 - H_2 atmosphere and slowly cooled, and b) in vacuum and rapidly cooled.

The microstructure of these samples after the industrial sintering (Fig.2a) consists mainly of bainitic areas, with some pearlite. In accordance with [9] some martensite was additionally identified, especially in areas with higher manganese concentrations. Sintering in a vacuum, together with rapid cooling determines a basically martensitic microstructure (Fig.2b).

The hardness and density of investigated materials are summarised in Table 2.

Tab.2. Hardness values (HV 10) and density of Fe-2Mn-0.85Mo-0.5C materials prepared under "industrial" and "laboratory" conditions.

| Property | Industrial sintering | Laboratory sintering |
|-------------------------------|----------------------|----------------------|
| Hardness HV10 | 170 | 430 |
| Density [g·cm ⁻³] | 6.89 | 7.12 |

The evolution of the friction coefficient was recorded during each dry sliding test carried out in a pin-on-disc configuration. The tests were performed under similar conditions except for the load applied on the pin. The applied load for industrial sintered samples was 5 N, while for laboratory sintered ones it was 15 N, as for 5 N the mass loss was too small for recording. The obtained friction coefficient traces revealed an irregular character (Fig.3).

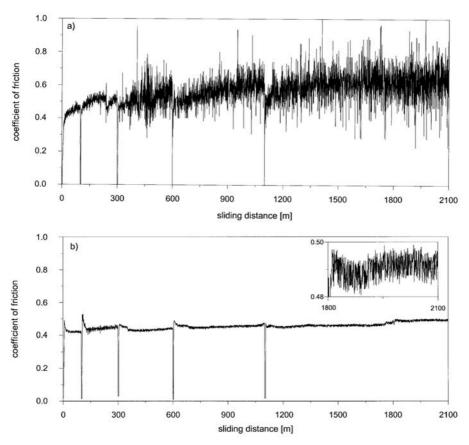


Fig.3. Evolution of friction coefficient during the wear tests: Fe-2Mn-0.85Mo-0.5C sintered under a) industrial and b) laboratory conditions. Insert in Figure (b) visualises an irregularity of the curve by presenting part of it on a different scale.

Each test was interrupted at defined intervals and discs were weighed to define the curve of cumulative mass losses (Fig.4)

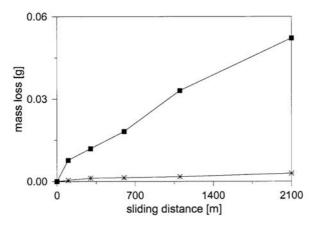


Fig.4. Mass losses during the wear test: Fe-2Mn-0.85Mo-0.5C sintered under industrial (squares) and laboratory (stars) conditions.

From Figure 4 it is clearly visible that the behaviour of the Fe-2Mn-0.85Mo-0.5C materials sintered under industrial conditions considerably differs from the behaviour of those sintered under laboratory conditions. A sensible increase of wear resistance is obtained with the application of rapid cooling, coupled with an increase in apparent hardness. The explanation for the above result has to be related to the microstructure of the material. The basic mechanisms of this behaviour are still under evaluation, but that problem is beyond the scope of this article.

As already mentioned above, friction coefficient traces revealed an irregular structure. In general, the measured fluctuations of the friction coefficient consist of real fluctuations of frictional force and a "noise" caused by a measuring device (due to the rigidity of apparatus, etc.). It can be assumed that the "noise" contribution of a particular device is nearly the same for the various materials being tested and for various experimental conditions. Therefore, the measured fluctuations of the friction coefficient can be used for qualitative characterization and comparison of various experimental results provided by the same device. But for a more detailed quantitative analysis and comparison of results from different devices, the properties of the measuring apparatus should be taken into account more carefully.

The recorded friction coefficient traces were processed by means of Higuchi's estimator. Typical results are summarised in Tab.3. One can see that the mass wear rate of an industrial sintered sample is much higher than that of a laboratory sintered one. The fractal dimension of the corresponding friction coefficient trace is higher too, but the difference is not so distinct. During an individual dry sliding test, the mass wear rate may also fluctuate at a steady-state stage, and the fractal dimension sometimes does not follow these changes. The discrepancy can be caused by an apparent ductile character of the samples. This enables a large release of accumulated stress through a plastic deformation in a general sense of this term, without removal of material away from the sample, that is, without change in sample mass.

| · · · · · · · · · · · · · · · · · · · | | • | | |
|---------------------------------------|--------------------------------|-----------|--------------------------------|-----------|
| | Industrial sintering | | Laboratory sintering | |
| Sliding distance interval [m] | Wear rate | Fractal | Wear rate | Fractal |
| | $[\times 10^{-6} \text{ g/m}]$ | dimension | $[\times 10^{-6} \text{ g/m}]$ | dimension |
| 300 – 600 | 21 | 1.9722 | 0.7 | 1.9387 |
| 600 - 1100 | 29.8 | 1.9892 | 0.6 | 1.9491 |
| 1100 - 2100 | 19 | 1.9890 | 1.2 | 1.9715 |
| 300 - 2100 | 22.3* | 1.9863* | 0.94* | 1.9598* |

Tab.3. Steady-state wear rates and fractal dimensions of friction coefficient traces for differently sintered Fe-2Mn-0.85Mo-0.5C materials. *average values.

To demonstrate the presence and role of plastic deformation, it is convenient to evaluate the volume wear rate too. The wear volume was determined by a careful analysis of the profile of the final wear track (Fig.5), and subsequently converted into a theoretical wear mass by means of material density. Unfortunately, only the total wear volume was determined, so only the total mass loss was calculated. The mass loss calculated from wear volume was higher than the mass loss actually measured (Tab.4). This indicates that a large amount of material was displaced from the wear track without removing it from the sample. So, a significant fraction of input power was consumed by an apparent plastic deformation. Hence, it seems that for materials capable of general plastic deformation, the mass wear rate is not a proper quantity which has to be correlated to the fractal dimension of friction coefficient trace. The volume wear rate looks more promising for this purpose.

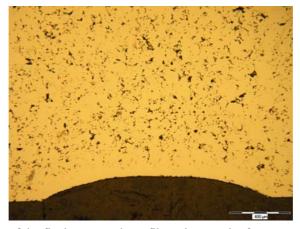


Fig.5. An example of the final wear track profile. Micrograph of a cross section through the worn surface upright to the wear track for Fe-2Mn-0.85Mo-0.5C material sintered under industrial conditions.

Tab.4. Overall wear characteristics of differently sintered Fe-2Mn-0.85Mo-0.5C materials.

| | Industrial sintering | Laboratory sintering |
|--|----------------------|----------------------|
| Total mass loss [mg] | 52.1 | 2.9 |
| Total volume removal × density [mg] | ~65 | ~3.8 |
| (Total volume removal × density)/Total | ~1.2 | ~1.3 |
| mass loss | | |

CONCLUSION

In this contribution, the applicability and usefulness of fractal geometry methods for the characterization of wear process taking place in materials capable for ductile fracture were investigated. The actual wear experiments were carried out in a pin-on-disc configuration with Fe-2Mn-0.85Mo-0.5C materials sintered under two different conditions: industrial (N_2 - H_2 atmosphere and slow cooling), and laboratory (vacuum and rapid cooling). The results of this preliminary study can be summarized as follows:

- 1. Application of Higuchi's estimator on the recorded friction coefficient data confirmed that the friction coefficient traces were fractal curves for sintered steels under investigation as well. But the effect of the "internal noise" introduced by a measuring device represents an open question. The problem is whether the tribometer used and pin-on-disc configuration are "rigid" enough (that is, their "internal noise" is quite small) to be applied for measuring the frictional force fluctuations. So, with respect to the further, more detailed, quantitative analysis, this question requires careful investigation.
- 2. There are indications toward the existence of a correlation between the wear rate and fractal dimension of friction coefficient traces, but not as distinct as it had been for the representatives of brittle materials. This can reflect the fact that the mass wear rate, measured in the experiments considered here, probably does not represent the proper wear rate in the case of materials capable of plastic deformation. For such samples, the material can be displaced from the wear track without removing it away from the sample, that is, without changing the sample mass. So it seems to be more suitable and more correct to measure the volume wear rate. This represents a goal for further experiments.

Acknowledgement

The authors are grateful to the Slovak Grant Agency for Science (Grant No. 2/3208/23) and CNR-SAS Project: "Microstructure and Mechanical Properties of Microand Macrograded Eco-friendly (Fe-Cr-Mn-Mo) porous materials" (2004-2006) for support of this work.

REFERENCES

- [1] MENG, HC., LUDEMA, KC.: Wear, vol. 181-183, 1995, p. 443
- [2] WILLIAMS, JA.: Wear, vol. 225-229, 1999, p. 1
- [3] KUPKOVÁ, M., KUPKA, M., RUDNAYOVÁ, E., DUSZA, J.: Wear, vol. 258, 2005, p. 1462
- [4] KUPKOVÁ, M., KUPKA, M., RUDNAYOVÁ, E., DUSZA, J.: Key Engineering Materials, in press
- [5] BERG, S.: Advances in Powder Metallurgy and Particulate Materials, 2001, p. 774
- [6] ENGSTROEM, U., et al. In: Proc. of PM²TEC 2002, part 13, June 2002, Orlando, USA, p. 1
- [7] ACTIS GRANDE, M. et al.: La Metallurgia Italiana, 2002, no. 9, p. 23
- [8] TAKANUR, SN., et al.: Int. Journal of Powder Metallurgy, vol. 40, 2004, no. 3,
- [9] DUDROVÁ, E., et al. In: Deformation and Fracture in Structural PM Materials. Vol.1. Stará Lesná, 15.-18.9.2002. Ed. Ľ. Parilák, H. Danninger. Košice: ÚMV SAV, 2002, p. 107
- [10] DUDROVÁ, E., KABÁTOVÁ, M., BIDULSKÝ, R., WRONSKI, AS.: Powder Metallurgy, vol. 47, 2004, p. 181
- [11] BOCCHINI, G., et al. In: Proc. of EUROMAT 2001, CD-ROM Edition, 10-14 June

- 2001, Rimini, Italy, ISBN 88-85298-39-7
- [12] ROSSO, M. et al. In: Deformation and Fracture in Structural PM Materials. Vol.1. Stará Lesná, 15.-18.9.2002. Ed. Ľ. Parilák, H. Danninger. Košice: ÚMV SAV, 2002, p. 38
- [13] CHAGNON, F., et al. In: Proc of PM²TEC '98. Vol. 1, Las Vegas, USA
- [14] ACTIS GRANDE, M., DUDROVÁ, E., KUPKOVÁ, M., KUPKA, M., ROSSO, M. In: 2004 PM World Congress & Exhibition. Vol. 2, October 2004, Vienna, Austria. Ed. H. Danninger, R. Ratzi, EPMA Shrewsbury 2004, p. 309
- [15] HIGUCHI, T.: Physica D, vol. 31, 1988, p. 277
- [16] SIMARD, P., LA TAVERNIER, E.: Mechanical Systems and Signal Processing, vol. 14, 2000, p. 459