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Assessment of wear behaviour of copper-based nanocomposite at the nanoscale

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ABSTRACT

Nanoscale wear behaviour of copper-based nanocomposite with Al_2O_3 nanoparticles have been investigated with the help of the circular mode atomic force microscopy (CM-AFM). The occurrence of running-in and steady-state wear regimes is similar to macroscopic behaviour described by Barwell. Archard's macroscopic wear equation, which states that the wear value is proportional to the applied load and independent on the sliding speed, is also valid at the nanoscale, with the limitation that the normal load should reach a threshold value to generate wear. Eventually, it is shown that the wear value at the nanoscale is highly dependent on the nature of the counter-body (AFM tip) material.

1. Introduction

Wear at nanoscale is a field of growing interest, due to the nanotechnology developments and their increasing applications in microelectromechanical and others systems. Experimental and theoretical studies of wear behaviour on macroscale are numerous, while the experimental difficulties hinder noticeably experimental studies on nanoscale. For instance, many friction experiments at nanoscale are described as operating in a "wearless" regime, i.e. with a wear rate considered below the detection limit of the experiment. Such low wear rates make it difficult to quantify wear [1]. In addition, there is a problem connected with the atomic force microscope (AFM) tip wear, which exist even when e.g. diamond tip is in contact with softer counter-body [2]. Furthermore, a complete 3D quantification of the wear volume is difficult at the atomic scale. Most of the investigations at the nanoscale reported in the literature are typically based on 2D images analysis of scanning electronic microscope (SEM) AFM tip images [3]. In this latter case, the worn volume is usually calculated by assuming that the tip worn is was cone shaped [4].

Phenomenological laws of dry sliding adhesive and abrasive wear of metals on macroscale were established by Archard, more than 60 years ago [5]. Among other relevant features, he has concluded that the wear rate is proportional to the applied load and is independent on sliding speed. In addition, Burwell and Strang [6] showed that the wear volume is proportional to the sliding distance, while Barwell [7] gave a set of three different empirical wear laws describing respectively the running-in, the steady-state, and the wear-out regimes. Nowadays, these wear regimes are presented through the wear curves, i.e. wear volume dependence on sliding distance [8]. Although broadly applicable for the macroscale wear behaviour, these underlying rules remain unclear at the nanoscale.

Many studies on nanoscale friction behaviour are available in the literature, while considerably less on nanoscale wear behaviour and wear quantifications in dry sliding conditions may be considered [1,2,4,9–12]. Further on, some of these studies were performed with constant load [2] and/or with constant sliding speed [1,2,9–12], and/or with constant or short sliding distance (shorter than 1 m), excluding them from the steady-state wear regime analysis [2,9,11,12]. Such a lack of data makes quit difficult a comprehensive full-scale qualitative and quantitative comparison of the results with that of macroscale wear laws. In addition, most of the studies were only investigating tip wear [1,4,9,11] and/or metalloids or non-metallic materials like silicon (Si), silicon nitride (Si_3N_4) and diamond [1,2,11,12]. As the last but not the least, the whole set of prior studies were performed on AFM with the

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linear (back and forth) scanning mode, which has sizeable drawbacks when compared with the circular mode AFM (CM-AFM) [13,14].

This paper reports wear behaviour in dry sliding conditions of a copper-based metal matrix nanocomposite (MMnC) at the nanoscale. This material used in industrial application is heterogeneous, rough and lightly sensitive to oxidation. Exploring wear on this kind of materials is challenging and more interesting for industrial relevance. The main goal of this investigation is to gain better understandings of the nanoscale wear behaviour for metallic-based composite materials. Specifically, wear dependence on sliding distance, sliding speed and normal load of relatively soft copper-based MMnC with Al_2O_3 nanoparticles in contact with AFM tips of two different materials (silicon nitride and diamond-like carbon), with relatively long sliding distances (up to 2600 mm) and a broad range of sliding speeds (up to 1.8 mm/s) were investigated with the CM-AFM.

2. Experimental details

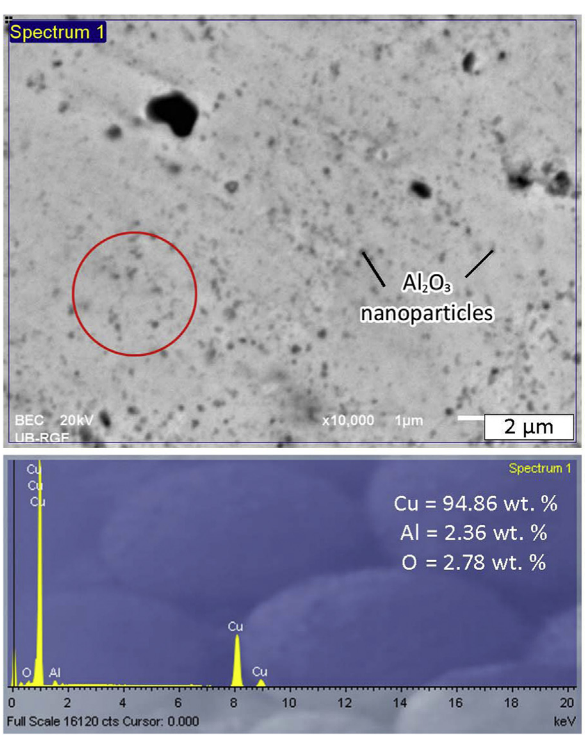
2.1. Tested material

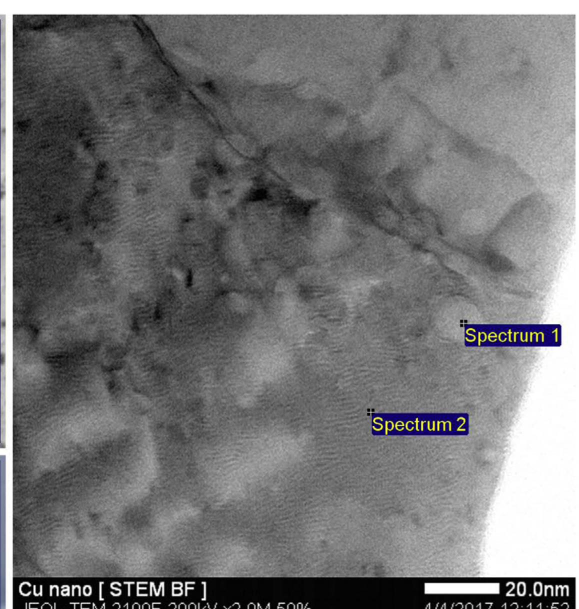
The copper-based MMnC with Al_2O_3 nanoparticles (referred to as nanocomposite) was used as model (sample) material. The average size of Al_2O_3 nanoparticles was less than 100 nm. It was produced by powder metallurgy technology (preceded by mechanical alloying and internal oxidation). The starting component was a prealloyed copper powder containing 2.5 wt% Al. This powder was subjected to internal oxidation, i.e. during milling, aluminium being less noble than copper, dissolves in the copper matrix, oxidizes first reacting with oxygen from the air and forms Al_2O_3 nanoparticles. Assuming that the complete amount of Al was oxidized, it was theoretically calculated that the amount of Al_2O_3 nanoparticles was 4.7 wt% [15]. Taking into account the densities of the MMnC phases, the theoretical volumetric amount of Al_2O_3 nanoparticles was about 10 vol%. Transmission electron microscope (TEM) analysis confirmed the presence of Al_2O_3 nanoparticles (right image on Fig. 1). The SEM image of the polished nanocomposite sample (left image on Fig. 1) shows the distribution of Al_2O_3 nanoparticles and, as a reference, the wear track size made by the AFM tip (with an approximate diameter of 2.8 µm). Additional energy-dispersive spectroscopy (EDS) analysis (Spectrum 1 on SEM image) of the whole surface confirmed the above mentioned weight amount of Al_2O_3 nanoparticles. It is important to notice that there is always a possibility that selected surface contain agglomerations of Al_2O_3 nanoparticles or that the agglomerations of Al_2O_3 nanoparticles were outside of the selected area. As a result, Al and O amounts in the EDS analysis would be respectively increased or decreased.

The samples were polished before the tests, and the initial arithmetic mean deviation of the assessed profile (*Ra*) and root mean square deviation of the assessed profile (*Rq*) of the surface of the samples (measured from 5 × 5 µm topographic AFM images), were Ra = 2.94 nm (SD = 0.56 nm) and Rq = 4.02 nm (SD = 0.85 nm). Microhardness of the samples was 245 HV 1 (SD = 11), with the average indentation diagonal of 87 µm. The copper-based MMnC was chosen as a relatively soft material in order to minimise the wear of the AFM tips.

2.2. Wear tests with the CM-AFM

Nanoscale wear experiments have been conducted with a DI-3100 Bruker AFM implemented with the circular mode (CM-AFM) [13]. The circular mode consists in imposing a circular displacement at high frequency (50 – 500 Hz), to an AFM tip in sliding contact with the tested material (Fig. 2). Contrary to commercial AFM scanning procedures, where the AFM tip is subject to a back and forth displacement with stop periods, the CM-AFM allows reaching a continuous relative displacement of the tip in contact with the surface at constant and high sliding speeds (up to 6 mm/s). As a result, it limits advantageously the drift of the piezoelectric actuator, in keeping a zero averaged voltage and a unique value of the frequency applied to the scanner. Performing experiments at a high sliding speed also reduces the duration of the experiment necessary to achieve high sliding distance (more than two and a half meters) in a reasonable time. The resulting circular wear track is easily distinguishable among intrinsic sample scratches and,





Element, wt.%	O	Al	Cu
Spectrum 1	12.16	8.74	79.10
Spectrum 2	0.05	0.89	99.06

Fig. 1. Polished surface of nanocomposite (SEM left and TEM right), and corresponding EDS analysis results; imaginary wear track is denoted with red circle of about 2.8 µm in diameter in SEM image.

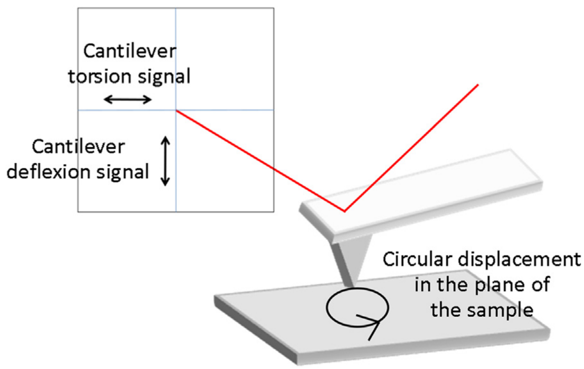


Fig. 2. Schematic diagram of nanoscale wear testing with the CM-AFM.

comparatively to the back and forth procedure, the CM-AFM allows exploring wear in all crystalline directions. In such conditions, wear tracks are clearly defined allowing the easy computation of the wear volume, even if the surface is not perfectly flat.

All wear values were obtained by wearing a fresh place on the sample, i.e. a new circle was generated for each wear value. The tip was not changed during the experiments, i.e. all silicon nitride/diamond-like carbon tip tests were performed with the same tip. The exceptions are connected with fracturing of some tips resorting to a new one whose tip radius is similar. Topographic images before and after wear were done in contact mode, with the unique AFM tip used for the experiments. After testing, worn surfaces of the AFM tips were examined using the SEM.

The AFM tips made of two different materials (silicon nitride and diamond-like carbon) were used in nanoscale wear experiments as counter-body, while the test samples were made of copper-based MMnC. Experiments were performed under dry sliding conditions, in ambient air, at room temperature (about 20 °C), with a relative humidity of 30%. In all experiments, the wear track was approximately 2.8 µm in diameter. The other testing conditions are summarised in Table 1, since they were different for silicon nitride (Si_3N_4) and diamond-like carbon (DLC) tip, as well as, for wear vs. sliding distance, wear vs. sliding speed, and wear vs. normal load tests. As an experimental precision, the normal load takes into account the adhesion force in the contact. This latter was determined by doing a force curve previously to the experiment. Two different tips were used in order to diminish the influence of potential tip wear, while the relatively long sliding distances in wear vs. sliding distance test were applied in order to get a steady-state wear. The Si3N4 tip had a 70 nm radius (spherical shape) with a 0.4 N/m stiffness of the triangular cantilever. On the other hand, the DLC tip had a 200 nm radius (spherical shape) with a 22 N/m stiffness of the rectangular cantilever. The tip radii were calculated from SEM images, while the cantilever stiffness values were determined by the thermal noise method [16].

Table 1
Testing conditions related to nanoscale wear experiments.

Tip material	Testing condition	Test		
		Wear vs. sliding distance	Wear vs. sliding speed	Wear vs. normal load
Silicon nitride (Si_3N_4)	Normal load	100 nN	–	70 – 200 nN
	Sliding speed	0.88 mm/s	–	0.88 mm/s
	Sliding distance	up to 2111 mm	–	106 mm
Diamond-like carbon (DLC)	Normal load	1 µN	1 µN	1 and 3 µN
	Sliding speed	0.88 mm/s	0.22 – 1.76 mm/s	0.88 mm/s
	Sliding distance	up to 2639 mm	317 mm	106 mm

2.3. Wear volume calculation

The wear volume quantification is challenging at the nanoscale, since the wear track is not regular. For each experiment, AFM topographic images in air, before (Fig. 3a) and after wear (Fig. 3b), for a given place, are subtracted to one another by using the specific homemade software to generate a difference image (Fig. 3c). By subtracting of the images before and after wear (Fig. 3c), the roughness of the unworn area is less than 0.2 nm. After that, average profile of the wear track is computed (Fig. 3d). The worn surface on the averaged profile is calculated in two ways, i.e. as a surface below the baseline determined by the software (dotted line on Fig. 3d) and as a surface below the manually drown line between the edges of the wear track on the average profile. In this way, two worn surface values are calculated for each wear track, which is denoted as min and max value. The calculated values are multiplied by the perimeter of the wear track to determine the wear volume values. Reported wear values are set as the average values of these minimum and maximum values.

3. Results and discussion

3.1. Wear vs. sliding distance

Calculated wear volumes for different sliding distances, i.e. wear curves, obtained with AFM tips made of two different materials at two different normal loads are shown in Fig. 4. These values in some way represent total wear values, since each of them were obtained by wearing a fresh place on the sample, i.e. a new circle was generated for each wear value. The tests were performed until the stabilisation of the wear occurs, i.e. until the steady-state regime is obtained.

The results confirm the trend which is common for the macroscale wear experiments, i.e. they confirm the occurrence of the running-in and beginning of the steady-state wear regimes at both applied normal loads, i.e. at 100 nN as well as at 1 µN (Fig. 4). Consequently, macroscopic wear law given by Barwell [7] hold on the nanoscopic length scale as well, at least for tested MMnC sample. The occurrence of running-in and steady-state wear regimes of silicon and silicon nitride AFM tips was already reported in the literature. In these studies, the steady-state regime was obtained with relatively long sliding distances (longer than 100 m) [1], as well as with very short sliding distances of 102 mm [11] and 4.5 µm [12]. On the other hand, according to our best knowledge, the existence of Barwell's running-in and steady-state regimes at the nanoscale, in wear of the softer material in contact (which is the case of the MMnC), was asserted only by theoretical modelling of two-body abrasive wear of Fe surface [17]. Moreover, in the study performed by Chung and Kim [9], the wear dependence on sliding distance for Cu, Au, DLC and bare Si sliding against a diamond-coated AFM tip, was mainly linear. In other words, there was no change of wear regime for any of the four tested materials. This could be due to the relatively short sliding distance of 200 µm, so the wear was probably still in running-in regime. Indeed, we do observe that the steady-state is reached for a sliding distance of approximately 1000 mm in our configuration (Fig. 4).

Analysing Fig. 4, it could be noticed that after onset of the steady-state regime, the wear values slightly decrease with the increase of the sliding distance, notably in the case of DLC AFM tip. This suggests possible wear of the AFM tips. However, SEM images of the AFM tips after the whole set of tests do not show visible wear. Also, self-tip imaging is not evidenced or features of the topographic images are not dilated due to a worn tip. Whatever, the AFM tip wear cannot be noticed and can be considered as negligible as compared to the wear volume of the sample that is an order of magnitude of 10^6 nm^3. Tested MMnC contains hard ceramic (Al_2O_3) nanoparticles which can

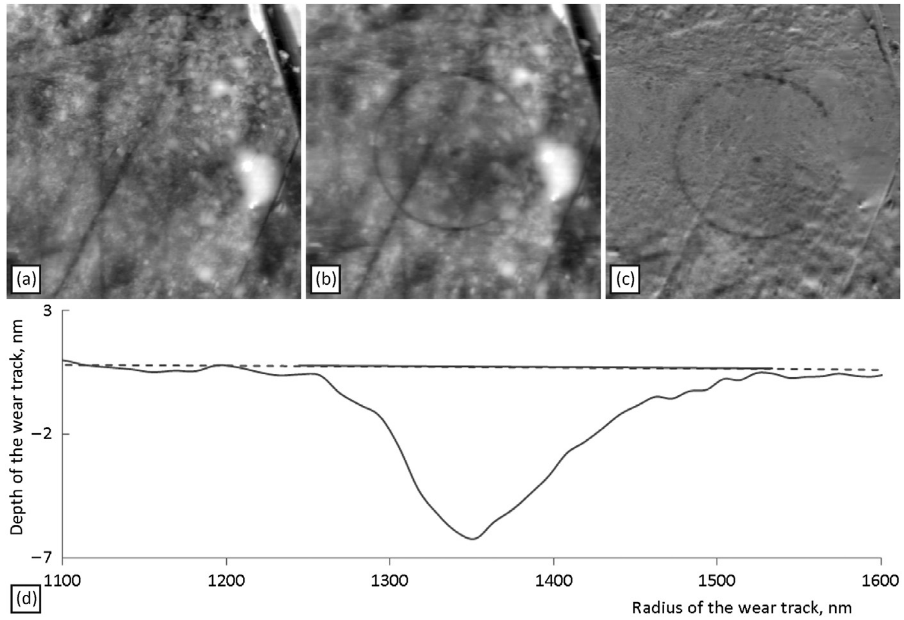


Fig. 3. Summary of the calculating steps to get the wear volume from the AFM topographic images: (a) AFM topographic image before wear experiment, (b) AFM topographic image after wear experiment, (c) subtracted AFM topographic image and (d) averaged profile of the wear track.

substantially influence localized high hardness. Since each of wear values were obtained by wearing a fresh place on the sample, it is normal to have the deviations of the results from the theoretical values. In addition, wear curve for tested MMnC in contact with DLC AFM tip (Fig. 4) suggests that we could achieve a steady-state for long sliding distance. Unfortunately, it was not possible to carry-on experiments at sliding distances longer than 2600 mm for this hard material (the tip breaks at very long sliding distances). Another interesting consideration is given by comparing the wear volumes generated by Si_3N_4 and by DLC tip (Fig. 4). The wear volumes are of the same order of magnitude, whereas the normal load is 10 times higher in the case of the experiments with the DLC tip. This observation highlights the role of the chemical or the physical-chemical nature of the counter-body material used in wear experiments at the nanoscale. Finally, even if the wear is more or less erratic, the behaviour is clearly defined.

Fig. 5 shows typical topographic AFM images of the wear tracks after imaging processing as described in the experimental section. The wear depths determined from the wear profiles from the images in Fig. 5 are in the nanometer range. These topographic images of the wear tracks generated by DLC tip (Fig. 5) show the real situation i.e. generated wear tracks look deeper and deeper, as the sliding distance increases. From Fig. 4, one can calculate in the steady-state regime for the DLC AFM tip that about 30 atoms of copper per micrometer of sliding have been removed. It is the double in the case of the Si_3N_4 AFM tip. Then an atom-by-atom or atomic cluster by atomic cluster or nanograin by nanograin removal process may be involved in the wear mechanism. Therefore, one might be aware that the observation of similar behaviours at the macroscale and nanoscale does not mean that the mechanisms involved in the wear process are similar.

3.2. Wear vs. sliding speed

Calculated wear volumes for different sliding speeds, obtained with a DLC AFM tip, are shown in Fig. 6. These values also represent total wear values, since the wear was measured at the end of each test. Even is the wear volume dependence on sliding speed is erratic, due to the heterogeneity of the material, the analysis of the data presented in Fig. 6 shows that most of the wear values are in the vicinity of the dotted horizontal line drown at the wear volume of 1.8×10^6 nm^3. This behaviour was also observed for high sliding speeds (equal and higher than 1 mm/s) by Bhushan and Kwak [4] in the case of dry and lubricated sliding contact with sputter deposited DLC film samples. These can be also noticed visually by comparing the topographic AFM images of the wear tracks generated at different sliding speeds (Fig. 7), since

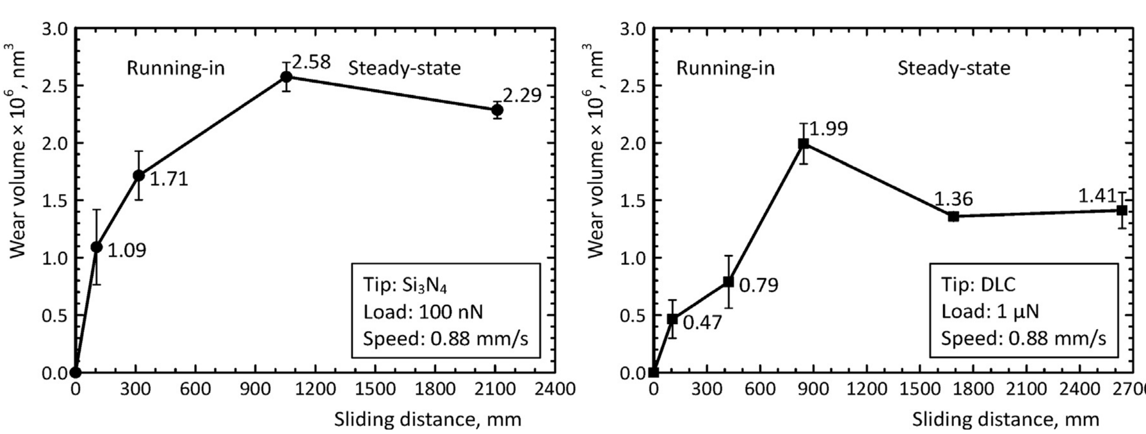


Fig. 4. Wear vs. sliding distance (wear curve) for tested MMnC in contact with Si_3N_4 and DLC AFM tip; error bars represent minimum and maximum values.

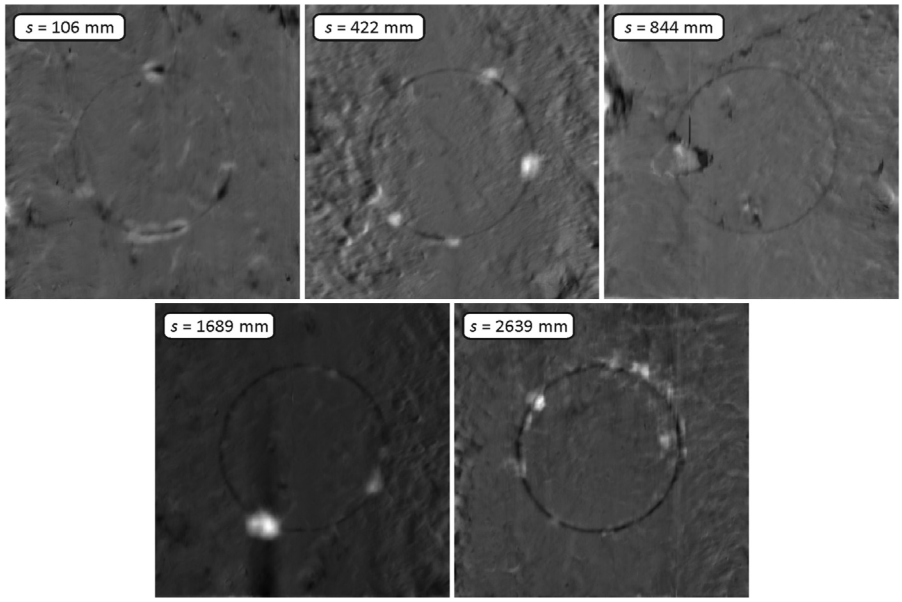


Fig. 5. Wear tracks (subtracted topographic AFM images; image size 5 × 5 μm) on tested MMnC for different sliding distances (s) generated by DLC AFM tip at normal load of 1 μN and sliding speed of 0.88 mm/s.

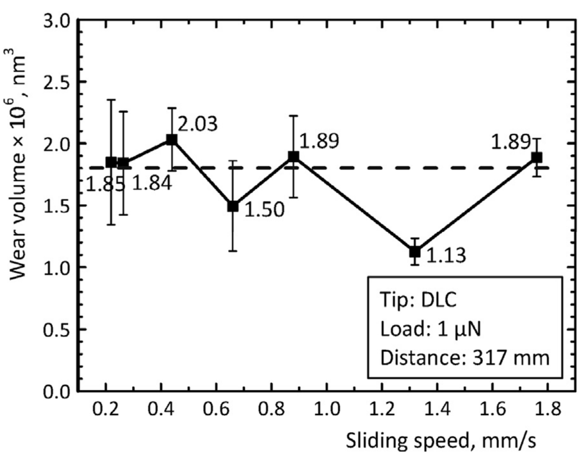


Fig. 6. Wear vs. sliding speed for tested MMnC in contact with DLC AFM tip; error bars represent minimum and maximum values.

the wear track depth look similar regardless the applied sliding speed.

3.3. Wear vs. normal load

The calculated wear volumes for different normal loads, obtained with AFM tips made of two different materials, are shown in Fig. 8. These values represent total wear values, after a relatively short sliding distance of 106 mm. This sliding distance was chosen in order to evaluate wear values in the running-in regime, i.e. before the onset of steady-state regime.

A first thing that could be noticed is that wear values obey Archard's wear equation for loads higher than 70 nN, i.e. that the wear value is proportional to the applied load. This trend can be clearly noticed on the corresponding topographic AFM images of the wear tracks after the tests (Fig. 9).

Since the wear track generated by the Si_3N_4 AFM tip at the lowest applied load of 70 nN is hardly visible, it is logical to assume that for lower loads wear could not be measured. This suggests that wear threshold may be considered, which in our case was 70 nN, i.e. a critical load value above which the wear value is considered proportional to the applied load. This could be associated with the wear mechanism change, considering the claim that as the load is increased (resulting in higher depth interactions), other wear mechanisms may become active, and that the chemical and structural composition of the surface interlayers could be substantially altered too [18]. Another explanation considers that the copper in air is covered by the oxide layer. The thickness of that layer depends upon time for exposure to air, and varies from 1.5 nm after approximately 20 min to e.g. 3 nm after approximately 30 h [19]. The copper oxide layer hardness is about 4 – 5 GPa, and it is about 0.5 – 1 GPa for the copper [20]. Therefore, the oxide layer exhibits a higher wear resistance than the pure metal. Considering a 70 nm Si_3N_4 tip with a modulus of elasticity in between 130 and 240 GPa, a load of 70 nN is exactly the load which generates a maximum shear stress during sliding at a depth from contact surface of approximately 3 nm. Accordingly, the threshold value of 70 nN may correspond to the load that develops a maximum shear stress at the oxide/metal interface.

The almost linear dependence of wear values from normal load for Cu coated silicon, as well as for Au, DLC and bare Si, sliding against a diamond-coated AFM tip is confirmed by Chung and Kim [9]. In their study, the total sliding distance was 200 μm (100 cycles of 2 μm scratch line-scan), and the applied load was varied from 100 to 800 nN, with a relatively low sliding speed (5 μm/s). By using the values they presented, an almost perfect linear fit can be constructed within the 200 – 800 nN loads range. The authors themselves acknowledge that for some specimens, the wear volume at relatively low loads was difficult to measure with confidence, so the associated data were not included in the results. A similar behaviour was obtained by other researchers, as well. Colaço [10] investigated stainless steel AISI 316 L wear in sliding contact with diamond tip, over the load ranges from 2 to 20 μN. The sliding distance was probably enough to obtain the steady-state wear regime (approx. 1 m), while the sliding speed was also relatively low (10 μm/s). He obtained well-defined worn craters only for normal loads of 8 μN or higher, whose dimensions increased with increasing loads. Hence, he assumed the relevance of a wear threshold after which the wear value is proportional to the applied load. However, this wear onset occurs for loads much higher than what can be expected from comparison of contact stresses with bulk hardness or yield stress.

A quite inviting feature could be noticed on Fig. 4, by comparing the wear values of MMnC in contact with Si_3N_4 and with DLC AFM tip. Tests with a DLC tip as counter-body were performed at loads that were

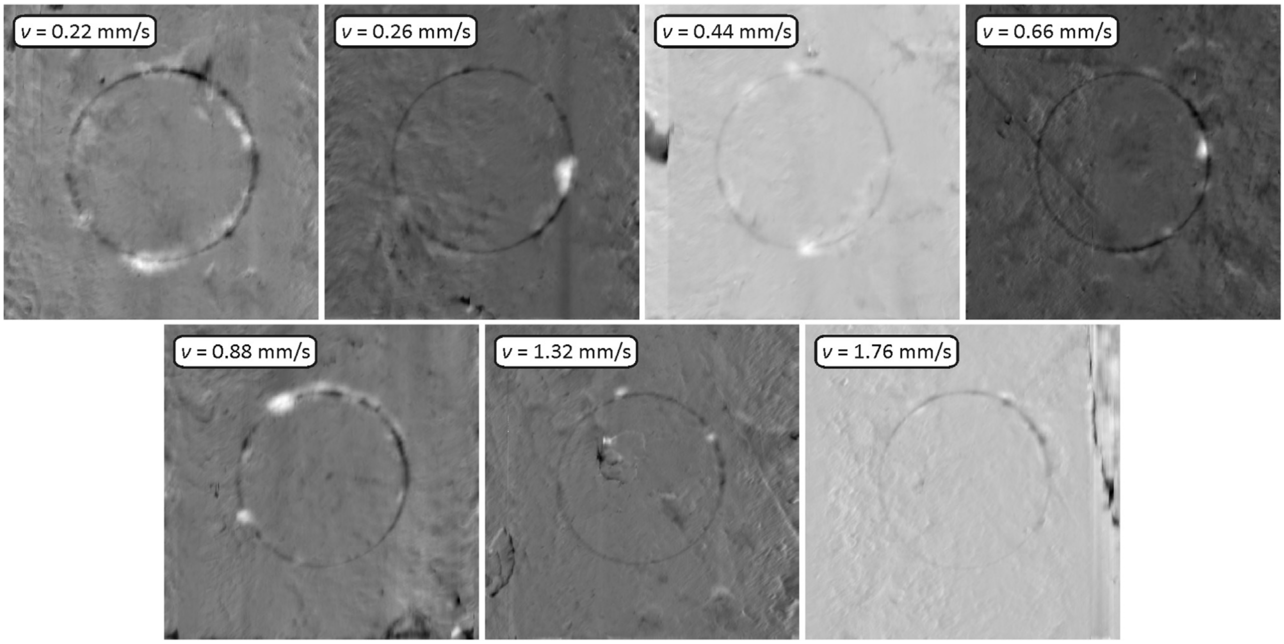


Fig. 7. Wear tracks (subtracted topographic AFM images; image size 5 × 5 μm) on tested MMnC for different sliding distances (*s*) generated by DLC AFM tip at normal load of 1 μN and sliding distance of 317 mm.

approximately one order of magnitude higher, and yet the wear values were similar or even lower than that in tests with the Si_3N_4 tip as counter-body. This is surprising, especially according to the fact that DLC is supposed to be harder than Si_3N_4. This could be due to the significantly different tip radius of Si_3N_4 tip (70 nm) and DLC tip (200 nm); since the Si_3N_4 tip is sharper it would induce a more intensive wear. Other reasons could also be associated with: different wear mechanisms operating on lower (Si_3N_4 tip tests) and higher (DLC tip tests) loads, MMnC inhomogeneous structure and particle distribution (Fig. 1) or adhesion and compatibility issues of the MMnC with its corresponding tip material. In addition, DLC is known to present very low friction, thus it is possible that the entailed low coefficient of friction in case of DLC tip tests induced lower shear stress and thus, lower wear values. Nevertheless, a comprehensive explanation of this phenomenon will be the subject of further research, and is beyond the scope of this specific study.

4. Conclusions

An experimental study of nanoscale wear behaviour of copper-based nanocomposite with Al_2O_3 nanoparticles is performed with an AFM implemented with the circular mode (CM-AFM) and counter-bodies of different material (Si_3N_4 and DLC AFM tips). Compared to commercial AFM modes, the methodology based on the CM-AFM gives well-defined wear tracks. Consequently, quantitative data of wear volumes are easily accessible.

The experimental results revealed the existence of a running-in and a steady-state wear regime, i.e. the validity of macroscopic Barwell's wear laws at the nanoscale. Macroscopic Archard's wear equation also looks valid at the nanoscale, since the wear value remains independent of sliding speed and proportional to the applied load. This study is the experimental evidence that macroscale wear behaviour may be recovered at the nanoscale in agreement with simulation recently reported in the literature. However, one might be aware that such similarities do not mean the wear mechanisms are similar and that this

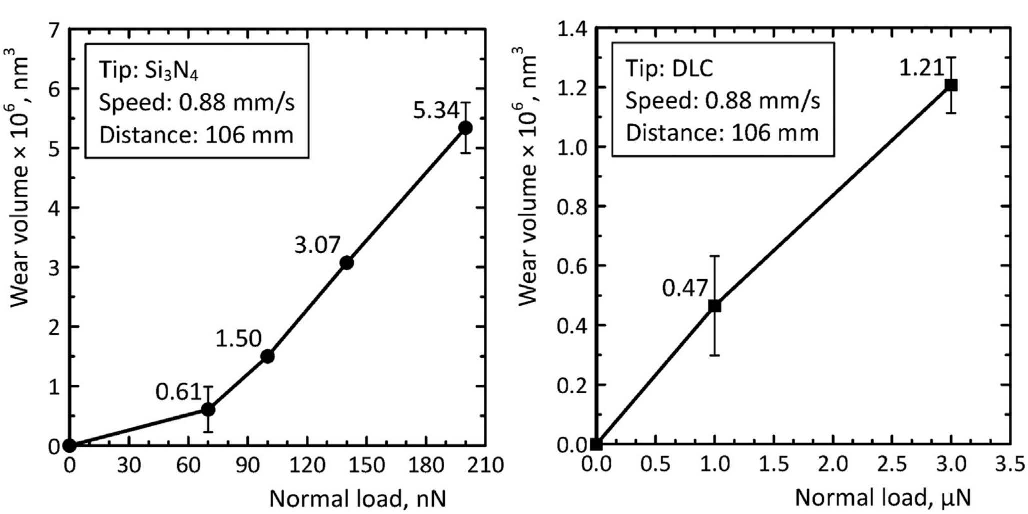


Fig. 8. Wear vs. normal load for tested MMnC in contact with: Si_3N_4 and DLC AFM tip; error bars represent minimum and maximum values.

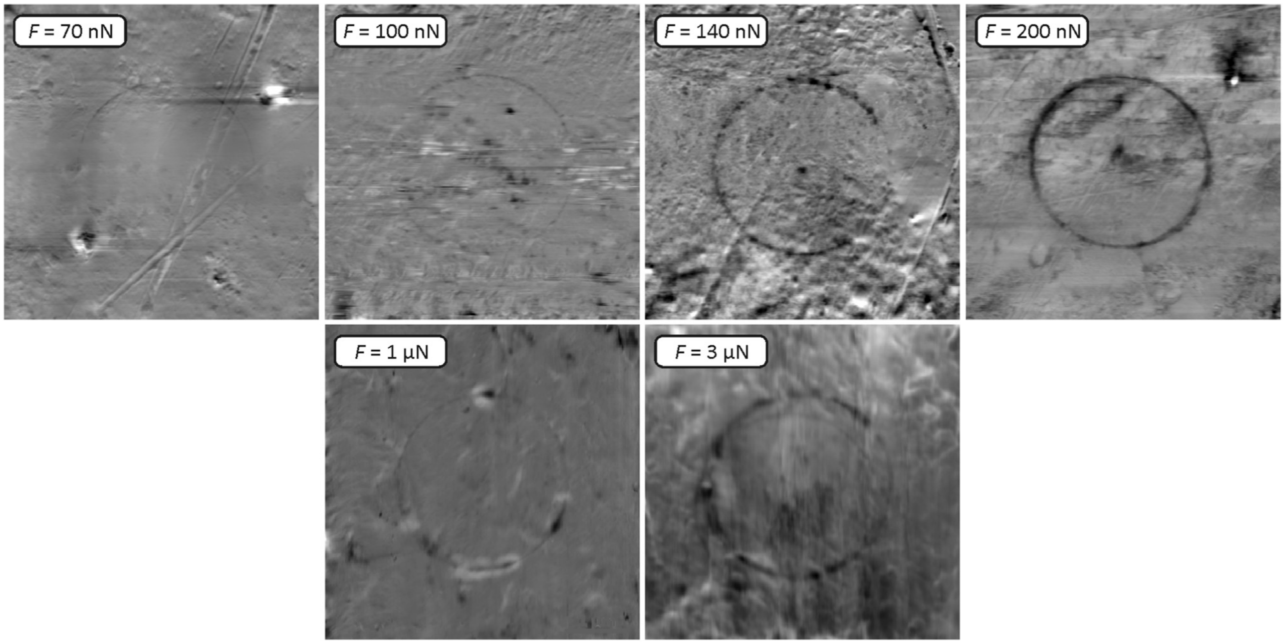


Fig. 9. Wear tracks (subtracted topographic AFM images; image size 5 × 5 µm) on tested MMnC for different normal loads (*F*) generated by Si_3N_4 and DLC AFM tip at sliding speed of 0.88 mm/s and sliding distance of 106 mm.

behaviour is universal at the nanoscale.

It is shown, for the used testing conditions and specific nanocomposite material, that there is a load threshold, which needs to be reached in order to have measurable wear. Eventually, it is also shown that the wear value at the nanoscale is highly dependent on the counterbody (AFM tip) material and geometry and physico-chemical interactions of the nanocomposite with AFM tip.

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