Effect of different types of graphene coatings on friction and wear performance of aluminum alloy

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Abstract

In this work graphene coatings have been deposited on aluminum alloy samples to investigate whether they can improve wear strength and promote friction reduction, which means components reliability and efficiency. Tribological performance has been tested by pin-on-disk wear tests. Three procedures to coat the samples have been explored. One is based on transferred graphene method and the other two are based on percolative graphene. Results show that graphene coatings last little time onto the sample surface, though strongly reducing the coefficient of friction of aluminum samples as far as they endure.

Keywords: graphene, coefficient of friction; aluminium; wear; 2D-material; nano-coatings;

1. Introduction

Every engineering interface undergoing relative movement suffers wear damage. This very general statement stands for both mechanical (structural) applications and electrical ones and, unfortunately, this is the case most of the times. Friction and wear can be reduced by lubricating the interface between sliding parts. Mechanical components are usually lubricated by liquid or semi-solid lubricants such as oils and grease. This remedy is often effective, though lubricants have to meet a lot of constraints of environmental compatibility, which do not concern only pollution. Fluid lubricants are critical in food industry for instance (as they should not contaminate food) and in high vacuum applications where very low volatility is imperative. As to electrical application, liquid and semi-solid lubricants may be undesired substances because of their electrical insulating properties, e.g. electrical connectors or switches.

For these reasons, solid lubricants and high lubricity coatings are very interesting for friction reduction as an alternative to fluid lubricants and they are already resorted to many fields of industry at present [1-5]. Low friction coatings have many advantages compared to fluid lubricants: notably they do not need any lubrication system (system design simplification), nor suffer leakages which basically takes away any pollution or contamination risk. Among the variety of materials used as friction reduction coatings, 2D-graphene layer has been investigated for the past few years by many authors in the scientific literature. Very interesting lubricant properties are attributed to graphene [6-8] and Geim and Novoselov at al. [9] and Wang et al [10] reported excellent mechanical, thermal and electrical properties for graphene.

Relatively large graphene sheets must be produced in order to coat mechanical or large electrical components. The production of large high-quality continuous graphene sheets is not an easy task, taking into account that the coating method must be reliable and cheap for industrial application. Two coating procedure are commonly addressed in the scientific literature to apply graphene onto a solid substrate, called transferred graphene and percolative graphene. Transferred graphene method [11] ensures higher quality graphene structure but, the production of large sheets is overly difficult and expensive. Percolative graphene [12], by contrast, allows for relatively large graphene sheets to be produced cheaply by piecing together graphene flakes. In this latter case the overall quality of the graphene structure may be poor and affected by flakes quality and dimensions.

As to tribological applications, graphene has been investigated as an additive for both liquid [13-16] and semi-solid [17, 18] lubricants, proving to be effective in reducing friction. Only a few investigations have been carried out so far where graphene is exploited as agent to improve the tribological behaviour of engineering materials in dry conditions, for instance on polymers [19], steel [20-22] and SiC [23].

In this paper, the tribological performance of a graphene coated aluminium alloy is explored by simplified tribological test. Aluminium alloys are increasingly used in structural applications due to their high mechanical strength coupled with a relatively low specific weight. Weight reduction in transportation industry is an increasingly crucial issue as part of the ongoing challenge towards energy consumption and emissions reduction. Aluminium alloys are indeed very promising materials in this field since the easiest way to achieve this goal is to design components made of low-weigh materials. These alloys find a large application as electrical conductor too, having a prominent electrical conductivity.

Plenty of examples of wear-related problems are available in the scientific literature concerning aluminium components. Wearing out has been reported for aluminium wires for electrical contact [24] and for tin plated aluminium connectors in transformers [25]. Structural parts of aircrafts and road vehicles are often fastened with bolts and rivets which are required to withstand high loads. Again, wear damage occurs at the interface of joined parts in the form of fretting, or else, because of repeated relative movement [26]. This kind of phenomena may be found also in oil drill pipe connections [27].

The aim of this investigation is to evaluate whether graphene coatings promise tribological-relevant benefits to envisage wider applications in both mechanical and electrical systems in the future, and to identify what kind of coating procedure may be more suitable for industrial applications.

Dry sliding tribological tests are performed by ball-on-disk method and the coefficient of friction (herein referred to as ‘CoF’) and wear rate of samples is recorded. CoF and wear rate of coated samples and bare samples are also compared in order to verify what is the most suitable coating procedure among those existing currently.

2. Materials and methods

The base material used in this work is an aluminum alloy, known with the trade name of Anticorodal (Al6082), whose chemical composition and mechanical properties are reported in Table 1. Samples were cut from a bar and coated with a graphene layer. Sample dimensions are about 12x12mm and 5mm thickness. Before the coating deposition, samples were polished and cleaned; the resulting average surface roughness after polishing is R_a 0.4μm.

Table 1. Technical specifications of the Al6082 aluminum alloy

Chemical composition – Al6082								
Cu	**Fe**	**Mn**	**Mg**	**Si**	**Zn**	**Cr**	**Ti**	**Al**
0.10	0.50	0.4 - 1	0.6 – 1.2	0.7 – 1.3	0.2	0.25	0.1	Balance

Mechanical properties – Al6082					
Tensile strength	Yield strength	Elongation at break	Specific weight	Elastic modulus	Hardness
275-310 MPa	230-260 MPa	4-10 mm	2.71 kg/dm^3	69 GPa	HB 83-94

The most promising currently existing methods to prepare graphene have been compared in this study. A total of three kind of graphene-coated aluminum alloy samples were tested: one sample coated with transferred graphene, one samples featuring percolative graphene with 3-hours sonication and one sample coated by 6-hour sonication percolative graphene. Details on the different methods are provided in sections 2.1 and 2.2.

Before coating processes, samples have been cleaned by sonicating 10 minutes with acetone and then drying by a lint-free tissue.

A pin-on-disk tribometer (Anton Paar TriTec, Corcelles, CH) was used to run unidirectional rotating tests against a 6mm 100Cr6 steel ball under a normal load of 5N (applied by means of dead-weight). The total sliding distance was 30m with a constant tangential velocity of 50mm/s and a sampling frequency up to 100Hz.

Tests were repeated two times with each sample at two different radii (2.3mm and 4.5mm) in order to account for the natural scattering of friction and wear phenomena. Samples dimensions prevented from running more than two wear tracks with each sample. Tribological tests were also performed on one more bare aluminum sample to have a benchmark for tribological performances.

Wear calculations were accomplished according to ISO 18535:2016 guidelines. Wear tracks were profiled at four locations perpendicular to one another.

2.1 Percolative graphene coating process

Graphene flakes are prepared by electrochemical exfoliation from a bulk graphite rod, using a platinum wire as grounded electrode [28]. Once the graphite rod is dissociated in flakes (applying 10V bias), they are collected by filtration and then dispersed in dimethylformamide. In order to get high-quality flakes and narrow the flakes thickness distribution, the obtained solution is centrifuged to remove side products and thick graphite particles. Centrifuged flakes are re-dispersed in added in ethanol with a mass concentration of 1 mg mL^{-1}. An Ultrathin Graphene Film (UGF), i.e. a continuous a graphene layer, takes shape by self-assembly process after dropping the obtained compound into DI water [12, 19]. The process is facilitated by sonicating this chemical solution with flakes floating on top. In this work, two sonication times are tried out: 3 hours and 6 hours. As shown by the Raman analysis (see paragraph 3.3), increasing the sonication time, the amount of graphene agglomerations increases too, since a larger number of flakes deform and roll up having time to clump together [29]. In both the cases graphene was transferred onto the sample surface by immersion directly into the DI water solution, like in water transferred printing techniques. Samples were finally dried at 75°C.

This process, resumed in Figure 1, creates a multi-layer graphene structure of about 2.5nm in thickness, corresponding to two or three atomic layers [12].

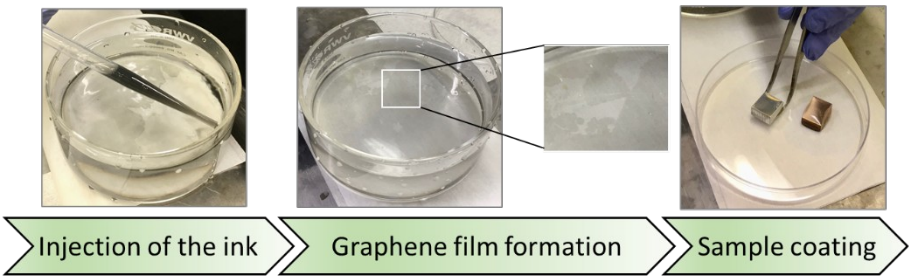

Figure 1. percolative graphene coating

2.2 Transferred graphene coating process

A layer of graphene film grows on a copper foil by Low Pressure Chemical Vapor Deposition (LPCVD) procedure [11, 21]. A layer of ethylene-vinyl-acetate (EVA) is then deposited onto the graphene layer as a support to make transfer onto the aluminum samples possible [29]. The copper foil is removed by etching and rinsed away with DI water. The remaining EVA/graphene layers are transferred onto the aluminum surface. Samples are heated at 80°C into nitrogen gas for one hour so that graphene grabs onto the metallic surface. The last step is removing EVA through xylene. By this method, resumed in Figure 2, the obtained graphene is a true atomic layer in thickness [30].

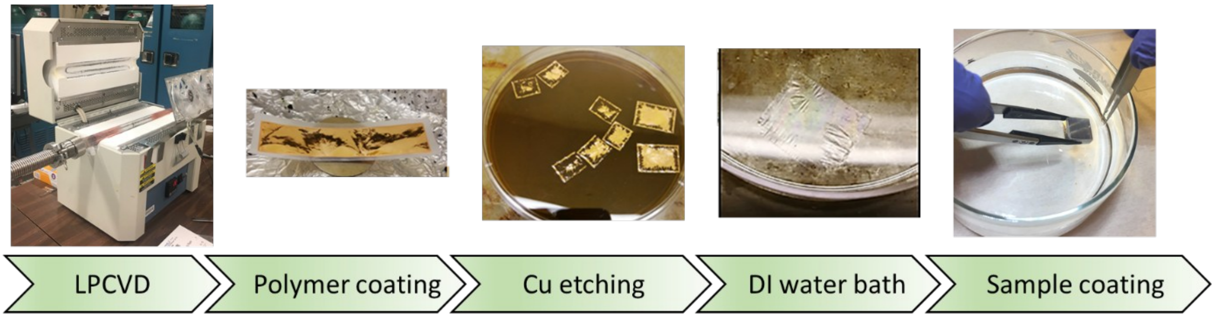

Figure 2. transferred graphene coating process

2.3 Spectroscopic Characterization by Raman Micro-Spectrometer

The three coated sample were characterized by means of a Renishaw InVia Reflex micro-Raman spectrometer (Renishaw plc, Wottonunder-Edge, UK), equipped with a cooled CCD camera. The Raman source was a diode laser (λ_{ex}=514.5nm), and samples inspection occurred through a microscope objective (20X), in backscattering light collection. 20mW laser power, 10 s of exposure time and 1 accumulation were employed to collect each spectrum. The system configuration allowed to focus different regions inside and outside the wear track, distinguishing thus still coated and areas where coating failed as a result of tribological surfaces interactions.

3. Results and discussion

3.1 Friction Coefficient Results

Figure 3 shows the four friction curves by pin-on-disc tests measured with the four samples presented in sec. 2 at 4.5mm radius.

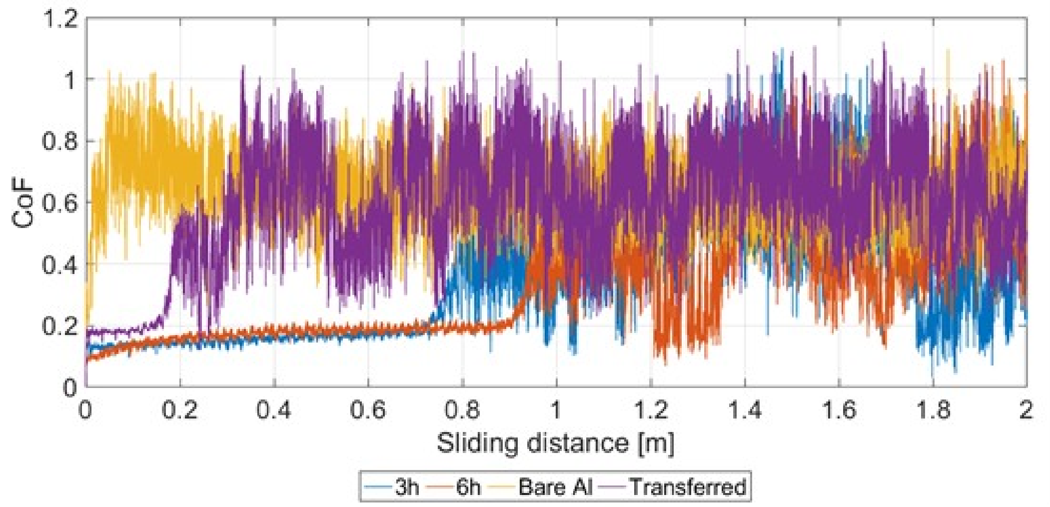

Figure 3. CoF trends against sliding distance (zooming the first 2m) at r=4.5mm

This diagram represents the first 2 meters of the tests only which is the significant sliding distance to understand the beneficial effect of the graphene coatings.

All the friction curves feature the same behavior outlined in Figure 4. In the first part of the test, coated samples show a relatively low CoF value with respect to the uncoated sample, and the curve trend is stable with low scattering of values too. The CoF increases then quickly after sliding a certain distance which varies from sample to sample though; huge scattering and unsteadiness of the signals arises after transition.

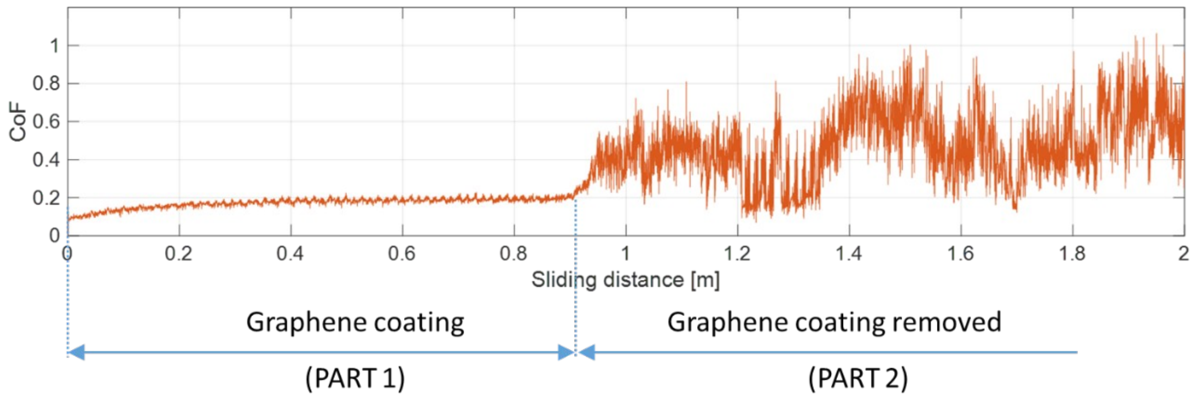

Figure 4. Example of C0F trend for coated sample: in Part 1 the graphene coating is working, in Part 2 graphene coating has been removed

This behavior is likely to be associated with the graphene coating removal from the sliding path. Evidence of that is the very similar behavior between the uncoated sample and the coated samples after CoF transition (see Figure 3 and Figure 4). Analysis by Raman spectroscopy presented in the next section confirms that no graphene or quite damaged graphene is found inside the wear tracks in all the samples. This confirms that the CoF transition coincides with the graphene coating failure.

The average CoF values of Part 1 and 2 (see Figure 4) are compared in Figure 5, together with the average CoF calculated over the whole test duration. The overall test average and the average related to the Part 2 are very similar indeed, because the Part 1 usually comes to an end within just few meters. Concerning the uncoated sample, the average CoF values calculated over the whole tests only, since it is not possible to distinguish Part 1 and 2 being no transition there.

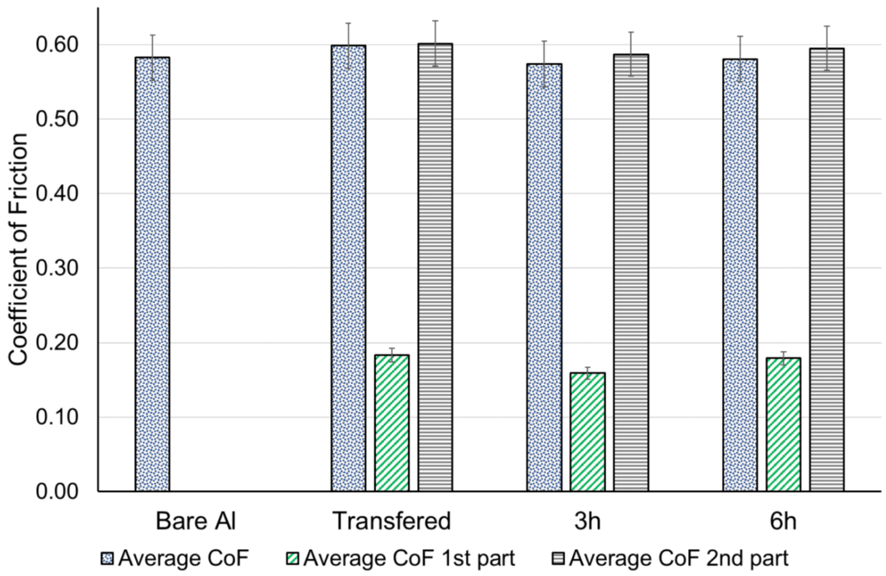

Figure 5. Average CoF values of the first and second part of the tests

Results show that graphene coatings can reduce friction a lot as far as they withstand the tearing-off effect of the sphere, with CoF dropping from about 0.6 to less than 0.2 for coated samples. More details and standard deviations values are resumed in Table 2. Moreover, the three graphene coatings show similar CoF values, the smallest value corresponds to the 3 hours sonicated coating, followed by the 6 hours sonicated coating and finally the transferred coating. The lower CoF in 3 hours sonicated coating respect to 6 hours sonication may be due to the GNP agglomerations (caused by the longer sonication time [29]) that have been appreciated by the Raman analysis results, described in paragraph 3.3.

Table 2. Average CoF values and respective statistical parameters

	Whole test		1st part		2nd part	
	Average CoF	**Standard Deviation**	**Average CoF**	**Standard Deviation**	**Average CoF**	**Standard Deviation**
Bare Al	0.583	0.030	N.A.	N.A.	N.A.	N.A.
Transfer	0.599	0.032	0.184	0.005	0.602	0.033
UGF 3h	0.574	0.007	0.159	0.016	0.587	0.013
UGF 6h	0.581	0.006	0.179	0.010	0.595	0.010

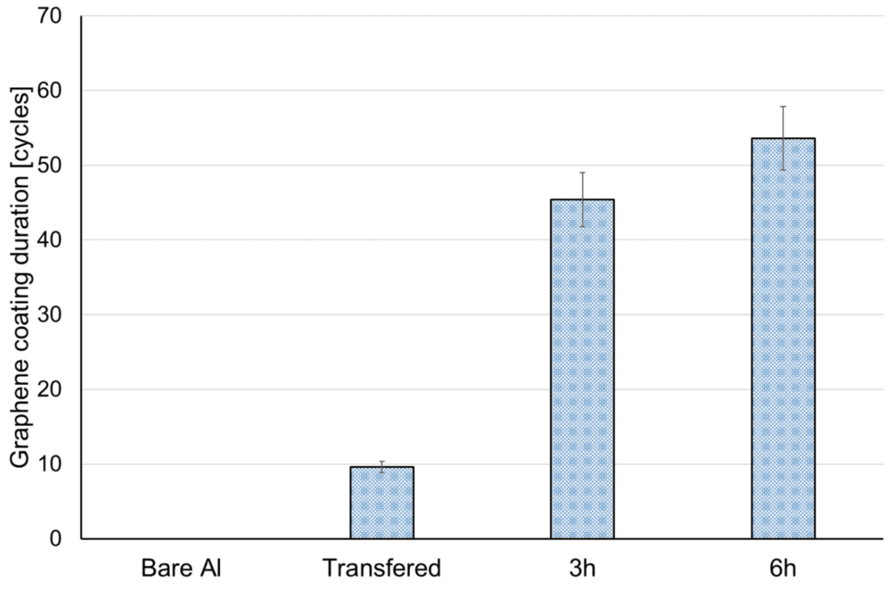

Figure 6. Average graphene coating lasting distance

Friction curves provide also information about the traveled distance before the coating failure, which is an estimate of the coating wear strength. In Figure 6, the average travelled distance at coating failure is shown for all the coated samples; in order to make an average within all the performed tests, the lasting endurance is now expressed in number of testing cycles lasted before the coating failure.

Results indicate that the strongest coatings are those prepared by the percolative method whereas the wear strength of transferred graphene is quite weak. This result can be explained by the fact that the quality of transferred graphene sheet is very high (see also sec.3.3) and its thickness is close to the atomic size. As to the percolative graphene, the UGF has coarse quality form the chemical point of view, which means that the coating structure is more likely to consist of several atomic layers. This results however in an enhanced wear strength.

The improvement of tribological performance due to graphene layer could be explained as 2D material as graphene inserted between contact surfaces allow reduction of shear stresses and therefore reduction of CoF [21, 30, 31, 32].

3.2 Wear tracks analysis

The volume of worn material. was calculated with reference to the method proposed by the ASTM G99 standard [33] as:

$$V = 2\pi \cdot r \cdot \overline{S} \qquad (1)$$

where *V* is the volume of the removed material, *r* is the nominal radius of the wear track and $\overline{S}$ is the area of the average cross section of the track. Wear tracks were profiled with a stylus profilometer (PGS200, SM Metrology System, Volpiano, IT) at four different locations at about 90° from one another. $\overline{S}$ is therefore calculated as the average of the cross-section areas laying underneath the four wear track profiles. Figure 7 resumes the calculated average wear volumes from each sample.

Being the sphere harder than the aluminum sample, minor damages appeared on it, being mostly limited to scratches. The sphere wearing-out was therefore neglected because the related wear volume was not measurable.

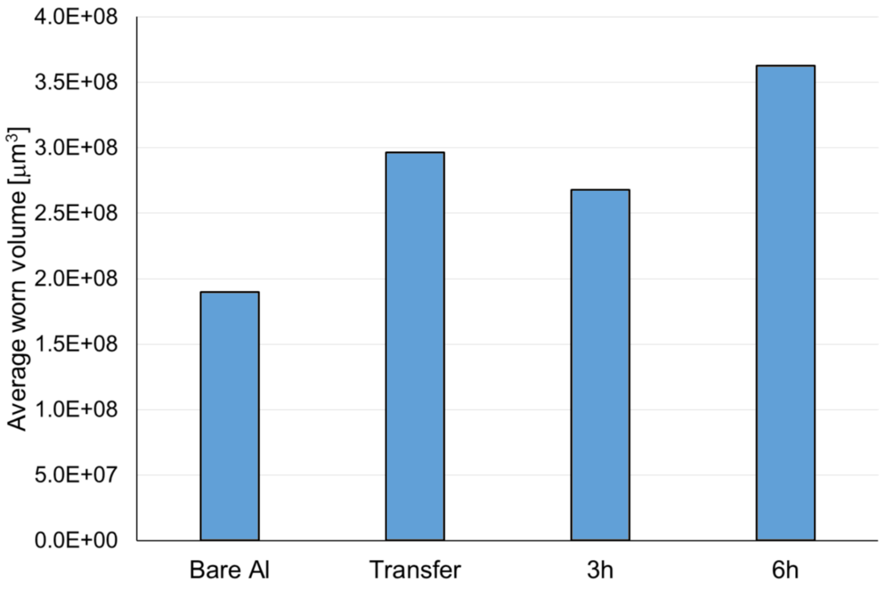

Figure 7. Average wear volume

Wear volume of coated samples is higher with respect to the uncoated sample. A possible explanation of this phenomenon could be that after graphene removal, the graphene particles mixed with aluminum debris act as abrasive bodies at the interface. In addition, it has to be considered that the wear volume is measured at the end of the test only, when the graphene coating is removed from the wear track (see also sec. 3.3). Provided that graphene has actual benefits on wear, this high-lubricity carbon sheet could not act but in the very first meters of tests. Friction curves in Figure 3 suggest in fact that it insulates the substrate as long as it holds onto the sample surface. Yet, any potential benefits vanish after transition. Graphene removal takes place too early in all the cases to appreciate an advantage in terms of total wear volume reduction over the whole test.

Analysis of wear tracks morphology was performed by SEM imaging. Figure 8 shows how the four wear tracks (at r=4.5mm) look like. The wear tracks of coated and bare samples are clear and outlined showing similar wear texture composed of several scratch lines along the sliding direction, indicating the most important wear form consists in abrasive wear.

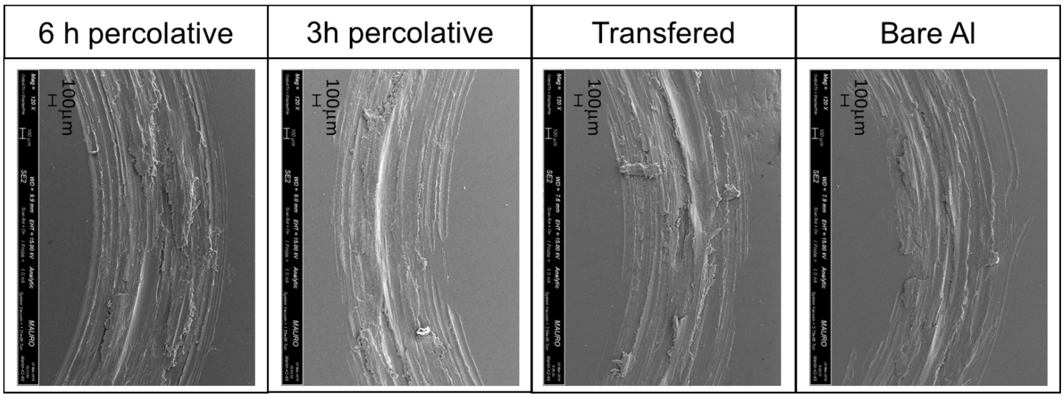

Figure 8. SEM images of wear tracks of the four kind of tested samples

Figure 9 shows the details of the SEM inspections of some debris found outside the wear tracks in coated samples. The first row of Figure 9 shows the debris, the areas identified by the red circles are then magnified in the second row, allowing to appreciate the presence of residual graphene layers attached to the debris.

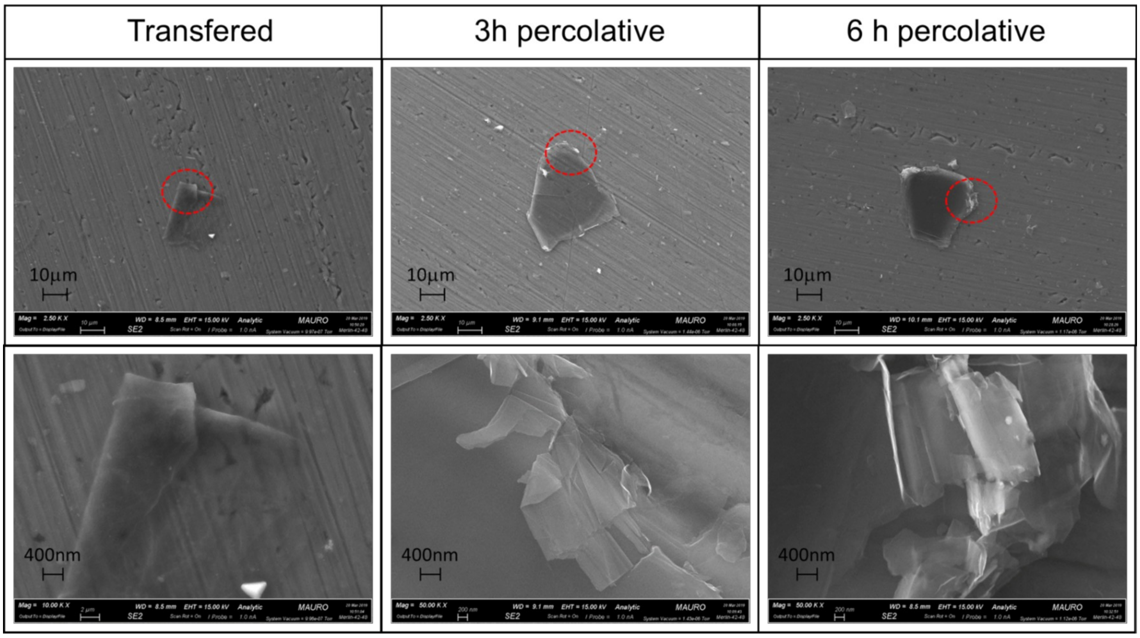

Figure 9. Details of debris found outside the wear track. It is possible to appreciate that the debris shows graphene coating

3.3 Characterization of graphene coatings

Graphene coatings have been characterized by SEM and Raman analysis, in order to check the coating quality, continuity and to analyses the graphene presence in the wear tracks.

Figure 10 shows the SEM images of the samples before the tests. Concerning coated samples, the transferred graphene coated sample shows uniform distribution of the coating (Figure 10 first row, second image). In percolative coatings (Figure 10 second row), it is possible to clearly identify the borders of the graphene nanoplatelets assembled. The coating results continuous, as later discusses also by the Raman analysis, in both 3h and 6h percolative coatings. In 6h percolative coating (Figure 10 second row, second image) some agglomerations are visible in the down left side of the image.

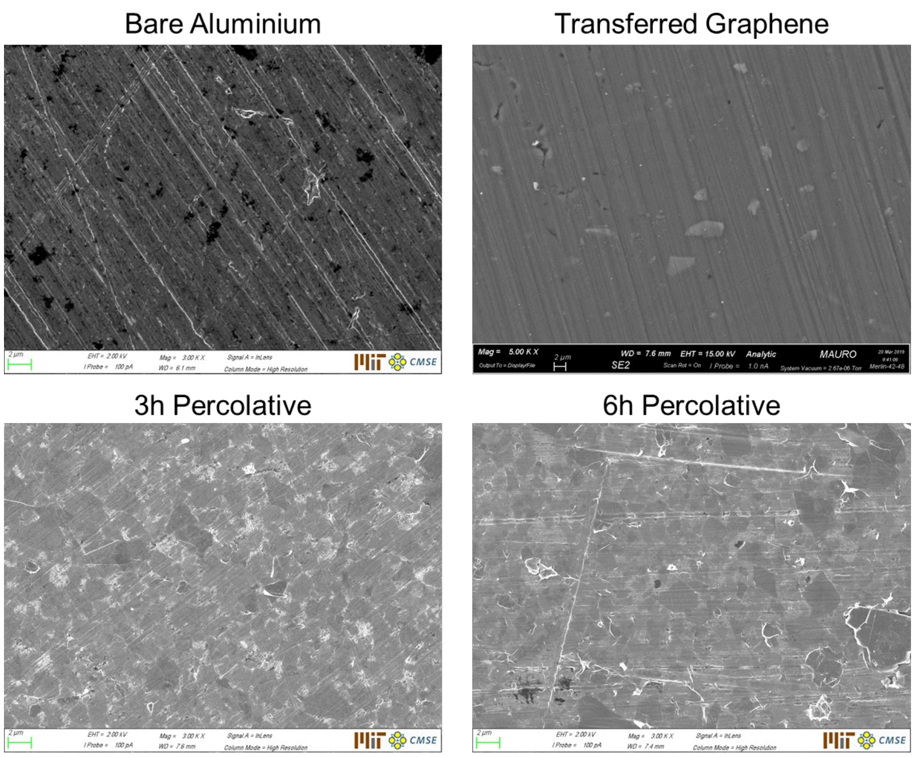

Figure 10. SEM images of the samples surfaces before tribological tests

Figure 11 reports Raman spectra collected at different regions of the three type of coated samples. As to the areas outside the wear tracks, the quality of LPCVD graphene (black line of Figure 11a) is quite high as the D peak at 1339 cm^{-1} is hardly discernible whereas the sharp G peak (at 1581 cm^{-1}), and 2D peak(at 2679 cm^{-1}) are intense and sharp. The integrated peak ratio I_{2D}/I_G ratio close to 3 confirms that a single layer graphene was obtained [28, 34]. Black lines in Figure 11 and Figure 11c are the typical spectra of graphene structures originated by flakes from electrochemical exfoliation of graphite followed by sonication [35]. Some additional peaks appear here: D, G, D', 2D, D+D' and 2D' peaks are positioned at 1350 cm^{-1}, 1583 cm^{-1}, 1614 cm^{-1}, 2694 cm^{-1}, 2932 cm^{-1} and 3187 cm^{-1}, respectively. The integrated peak ratio for 2D over G bands are coherent, for both the 3h-sonicated and 6h-sonicated UGF, which suggests a few-layered structure deposited on the Al samples.

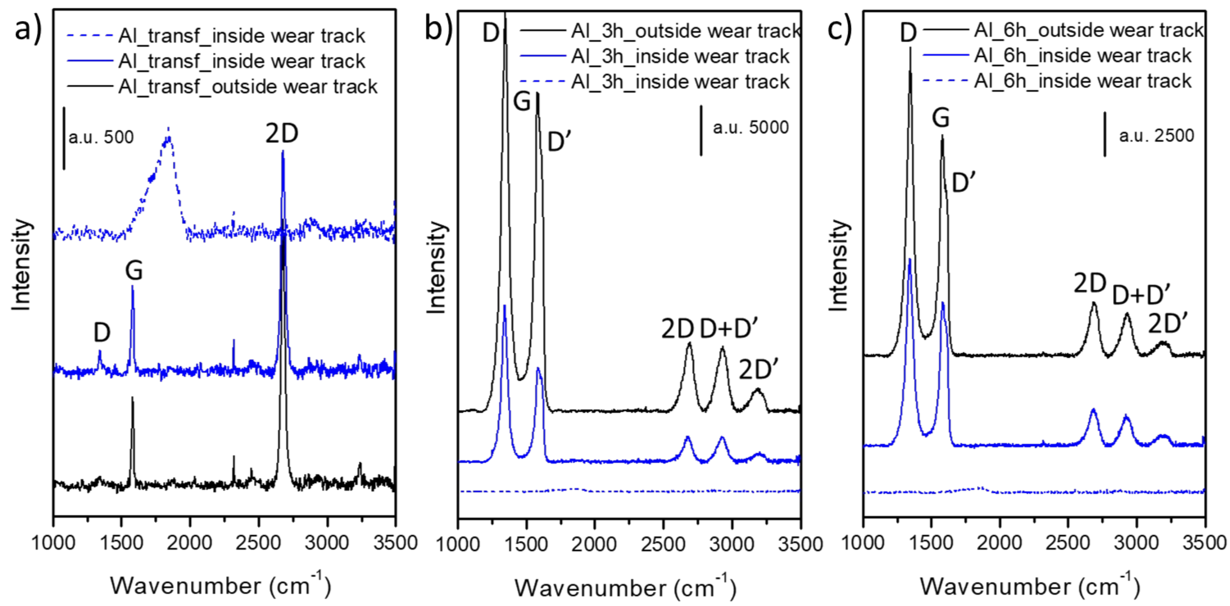

Figure 11. Raman spectra collected on a) the sample where LPCVD graphene was transferred on Al b) the Al sample coated by graphene deposited by means of the 3h percolative method c) the Al sample coated by graphene deposited by means of the 6h percolative method

Raman spectra analysis inside the wear tracks allows to evaluate the effects of wear tests. The removal of the graphene coating is indeed quite inhomogeneous inside wear tracks, blue solid lines represents the vibrational modes of areas graphene resists, i.e. remains of a graphene structure still exists even if the layers are more defective than the ones lying outside the wear track. This effect is more evident for LPCVD graphene sample (Figure 11a), as the D peak at 1339 cm^{-1} grows in intensity. Where the graphene failed (blue-dashed lines) a signal due to amorphous carbon [36] is observed and is particularly significant for the LPCVD-coated sample (Figure 11a), in accordance with previously discussed measurements.

Results in Figure 11 ensures that graphene is present outside the wear tracks for all the samples. In order to confirm the uniformity of aluminum coverage, a combined spectroscopic and optical evaluation was performed by sampling the test surface along a straight line close to wear track, each 40 μm. Such analysis has been possible thanks to the camera coupled to the Raman spectrophotometer microscope through a 20X objective.

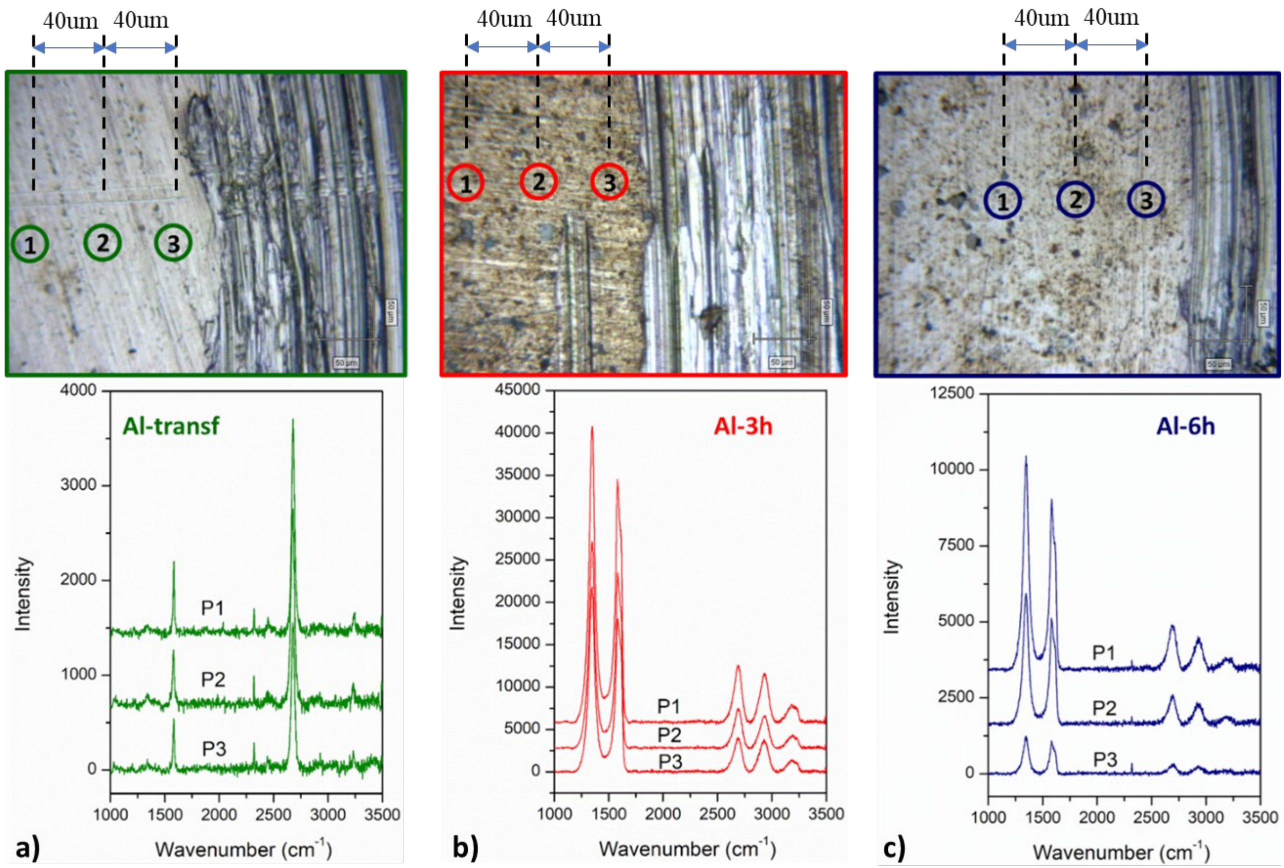

Figure 12. Optical images (top) and Raman spectra (bottom) collected along a straight line on different positions of the surface of the three coated samples, with a sampling frequency of 40 μm): a) Al with transferred CVD graphene; Al coated by graphene deposited through the flakes sonication procedure lasting b) 3h and c) 6h

Figure 12 accounts for the Raman spectra (bottom of image) collected in 3 different points (P1, P2 and P3) outside the wear tracks for each kind of substrate. Investigated areas are visualized on the optical images (top of the figure) and highlighted by circles. The corresponding spectra are qualitative similar each other for every kind of sample thus providing evidence of a good coverage uniformity. If some inhomogeneity in surface coverage would be present, the peaks shape and position should vary, and thus it should be revealed by comparing the spectra acquired on the same kind of sample, but in different position along the selected straight line. In addition, as a variation in peaks intensity is observable in particular for the spectra of the 6h-sonicated UGF (Figure 12c), a higher variability in thickness of the multi-layered graphene structure due to an accumulation phenomenon, on the Al surface is conceivable.

4. Conclusions

In this work, the tribological performance of graphene-coated aluminium alloy has been investigated. Three types of carbon nano-coatings obtained by transferred and percolative graphene were tested and compared. The nature and quality of the three types of graphene structures were successfully investigated by means of Raman spectroscopy and their coverage uniformity of the substrate confirmed. Tribological results show that a graphene coating is able to reduce the coefficient of friction up to 72%. Nevertheless, some critical aspects may make it difficult to use graphene extensively in mechanical and electrical applications in industry. The main issue is the relative low endurance under tearing-off action of a counter body and as soon as the graphene coating is removed from the surface, its benefit disappears. Among the other coatings tested, the longest-lasting and best performing one for tribological purposes turned out to be the percolative graphene prepared by 6h-sonication of flakes

As to wear, graphene coatings seem not to improve the wear properties of the system. This aspect should be further investigated in the future, however, as wear evaluations were carried out at the end of 30m lasting tests only. In all the cases, the graphene layer had been removed for a long time after such a long sliding duration. It would be worthwhile to explore the effects of graphene on the wear performance as long as the coating still separates the substrate from the counter body in the future.

Percolative graphene coatings are the most promising from the point of view of prospective industrial applications. They are easy to apply onto solid surfaces and show the greatest CoF reduction, lasting a relatively long time too. Therefore, even if UGFs from percolative methods are not as pure and high-quality as transferred graphene sheets, they would be much cheaper for emerging usage. Taking into account the weak strength of the coatings, prospective industrial applications would involve friction partners having a limited rubbing motion in time or amplitude; for instance, in oscillating bearings, electrical contact subjected to fretting damage and as in metal forming operations.

5. Aknowledgements

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