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Temperature-dependent contact phenomena of PVD- and CVD-deposited DLC films sliding on the thin aluminium foil

**Maria J. Díaz de Cerio · Gonzalo G. Fuentes ·
Rosario Martínez · Rafael J. Rodriguez · Elliott Spain ·
Jonathan Housden · Yi Qin · Wolfgang Hörnig**

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Abstract This paper reports on tribological properties of magnetron-sputtered WC–C and chemical vapour-deposited diamond-like carbon films coated onto hard-metal surfaces when sliding on aluminium foil (0.2 mm nominal thickness) at different temperatures. The study addresses the evolution of the coefficient of friction at the interfaces of the coated hard metal and the aluminium foil under dry-lubrication conditions, in a ball-on-disc configuration. The wear mechanisms of the aluminium foil and the damage produced on the coated surfaces due to the sticking of aluminium were evaluated as a function of the deposited coating and the temperature at their interfaces. Aluminium-transfer to WC–C coated hard-metal surfaces during the sliding operation seemed to be a non-continuous process, which appeared after a certain number of sliding cycles. Temperatures above 70°C accelerated the transfer of aluminium to the WC–C tool surfaces. Chemical vapour-deposited diamond-like carbon films hindered the transfer of aluminium to the hard metal even at temperatures of around 125°C. At greater temperatures, an aluminium–aluminium tribosurface is formed at the interface, which increases the wear rate of the foils and rapidly degrades the quality of coatings of the hard-metal surfaces.

Keywords DLC · Aluminium transfer · Tribology · Micro-forming

1 Introduction

The increasing demand of miniature/micro-products has led many researchers to investigate new processing technologies such as micro-forming and/or micro-machining [1, 2]. Besides the technological challenges associated with such complex processes, the sticking of the forming material onto the tool surfaces remains as a common problem associated to the manufacturing of small-size components. In the case of aluminium and its alloys, this feature becomes even more relevant due to its ductile properties at medium temperatures, leading to large adhesive forces during those operations where friction is present. Several authors have reported on this issue [3, 4], and different strategies have been used to overcome it [5–7], such as the optimisation of the cooling or the sliding contact speed.

Physical (PVD) and chemical vapour deposition (CVD) are universal coating techniques used for the surface

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M. J. Díaz de Cerio · G. G. Fuentes (✉) · R. Martínez ·
R. J. Rodriguez
Centre of Advanced Surface Engineering-AIN,
San Cosme y San Damian s/n,
31191 Pamplona, Spain
e-mail: gfuentes@ain.es

E. Spain · J. Housden
Tecvac Ltd,
Buckingway Business Park, Swavesey,
Cambridge CB4 5UG, UK

Y. Qin
Department of Design,
Manufacture and Engineering Management,
University of Strathclyde,
Glasgow G1 1XJ, UK

W. Hörnig
BPE Gmbh,
Föhrenstraße 51,
90542 Eckental, Germany

protection and enhancement of tooling surfaces [8–17]. Its effectiveness in protecting machining and forming tools at different conditions of contact load, wear, temperature etc. has been established. Magnetron-sputtered WC–C belongs to a group of materials, the so-called tribological coatings, having low friction and therefore a low tendency to pick up working material [9–11]. Diamond-like carbon (DLC) films as deposited using plasma-assisted CVD techniques emerge as feasible alternative coatings exhibiting very low friction: This occurs when their surfaces are saturated with C–H bonds [12].

These types of carbonaceous films have been investigated due to their capacity to prevent galling during different sliding-contact configurations, e.g. cylinder-on-cylinder [10], ball-on-disc [13]. The anti-galling properties of WC–C films were also reported in tribological studies involving AISI304 austenitic steels [10], dip-Zn-coated steel strips [13] and 100Cr6 bearing steels [14], as counter-face materials. However, few attempts have been reported when the counter surface of the tool/metal is aluminium in the form of thin foil. In this scenario, DLC films were investigated as potential solid-lubricant coatings for aluminium extrusion dies, showing good anti-sticking properties under oil-lubricated conditions [15]. Berger and Hogmark [16] reported that TiB_2-sputtered coatings also show a low tendency to pick up aluminium, using cylinder-on-cylinder tribological tests. More recently, Fuentes et al. [17] reported on the temperature-dependent tribological properties of WC–C films on aluminium foils, evidencing certain correlations between material pickup effects and temperature during sliding in a ball-on-disc configuration.

This work is actually a follow-up study of [17]. In this present study, a comparative investigation of the tribological properties of uncoated magnetron sputtering WC–C-coated and plasma-activated CVD (PACVD) diamond-like carbon-coated carbide balls sliding on thin aluminium foil is carried out using ball-on-disc configurations at different test temperatures. The results show that in order to prevent the galling of aluminium on hard metal, PACVD diamond-like carbon films outperform WC–C coatings deposited by sputtering techniques.

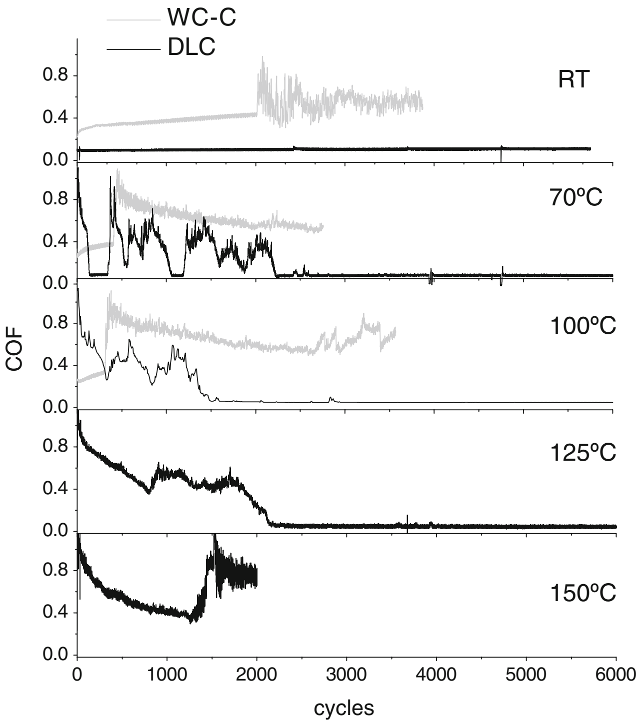

Fig. 1 Evolution of the COF of DLC- (*black*) and WC–C (grey)-coated carbide balls for different temperatures: RT, 70°C, 100°C, 125°C and 150°C, as labelled

2 Experiments

2.1 Materials and coating processes

Cold-rolled aluminium (99.5% purity from BPE Ltd.) in the form of thin foil (0.2 mm thick, $R_a = 0.42$ µm, $R_q = 0.53$ µm) was tested to monitor its tribological behaviour upon contact sliding against hard-metal balls of 6.3 mm φ, with a nominal composition in weight percent—92.7 WC, 1.0 TaC, 0.3 Cr_2C_3 and 6.0 Co. WC–C films were deposited by DC magnetron sputtering in an industrial deposition chamber at Tecvac Ltd (UK) with a base pressure of 1×10^{-6} mbar. The DLC coatings were prepared by PACVD using a standard commercial process at Tecvac Ltd. The maximum coating temperature did not exceed

Table 1 Coefficient of friction, universal hardness and reduced Young's modulus at 10 mN final load and wear coefficient (k_{wear}) of magnetron-sputtered WC–C and PACVD-deposited DLC films

Coating	Coatings parameters			
	Coefficient of friction[a]	HU (N/mm^2)	E' (GPa)	k_{wear} (m^3/Nm) × 10^{-16}
MS-WC–C	0.2	3,470 ± 150	79 ± 2	3.6 ± 0.3
PACVD DLC	0.1	8,800 ± 1,000	193 ± 5	0.52 ± 0.04

COF coefficient of friction, *HU* universal hardness, *E'* Young's modulus

[a] These values of the friction coefficient were measured using standard conditions for pin-on-disc tests (100Cr6 steel counterball 1/2 in./913.7 mm and 2 N load)

300°C. All processes included 5–10 min of substrate pre-etching in order to remove surface contaminants.

2.2 Coating characterisation techniques

The universal Vickers hardness (HU) of the deposited coatings was measured using an ultra-micro-hardness tester (Fischerscope H100XY VP) with a final load of 10 mN. The coefficient of friction (COF) and wear rates of the investigated films (Table 1) were determined using a CSM THT 8-153 tribometer employing a ball-on-disc configuration which permits a fine control of the temperature [17].

The tribological properties of all of the coated carbide balls on aluminium foil (0.2 mm thick) were monitored using the same ball-on-disc equipment/configuration under an applied load of 1 N during 2,000 cycles, with a sliding linear speed of 2 cm/s, to avoid breaking of the Al foil. Under these sliding conditions, the Hertz contact pressures yielded 490 MPa for the different coating/tool/foil combinations. In addition, the ball-on-disc tests were carried out on the coated hard-metal balls at constant temperatures of between 50°C to 150°C, using the same sliding parameters. The contact surfaces were inspected using a field emission scanning electron microscope (HITACHI S-4800), equipped with a secondary electron detector and energy dispersive spectroscopy (EDS) for elemental analysis.

3 Results

3.1 Standard characterisation of the films

Table 1 gathers the mechanical and tribological properties of the WC–C and DLC coatings as measured by ultra-

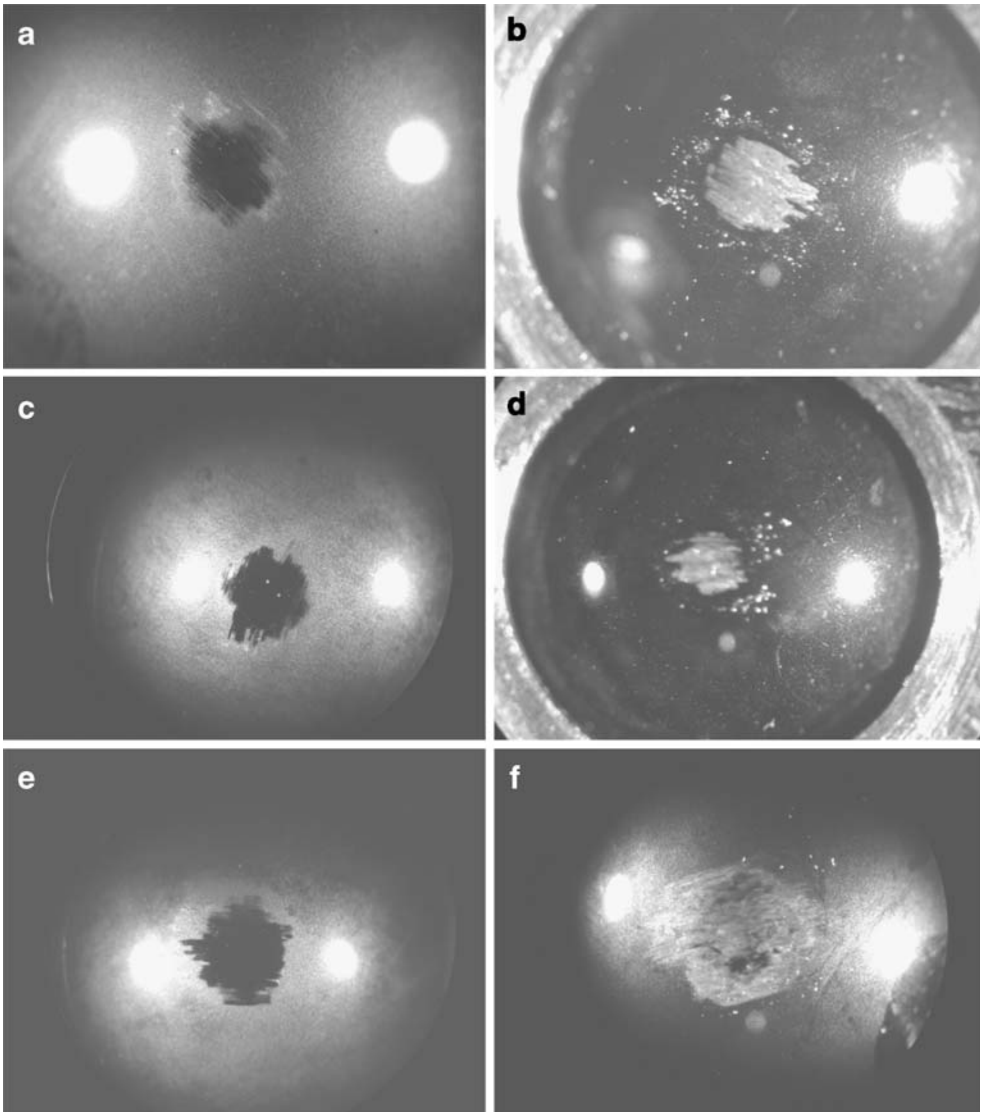

Fig. 2 Optical images (×40) of DLC-coated hard-metal contact areas after 10,000 cycles sliding on aluminium at: **a** RT, **c** 100°C, **e** 125°C and **f** after 200 cycles at 150°C. WC–C-coated hard metal contact areas after **b** 3,000 cycles at RT and **d** 1,000 cycles at 1,000°C

micro-hardness and standard ball-on-disc tests ASTM G99-05. The HU values for the WC–C film yields 3,440 N/mm^2, and its COF is 0.2 against 100Cr6 steel. The wear rates (k_{wear}) of the WC–C coatings were as low as 3.6×10^{-16} m^3/Nm. Additional data on the basic properties of other WC–C coatings can be found elsewhere [9]. The DLCs exhibited HU values of 8,900 N/mm^2 and wear rates one order of magnitude lesser than these of WC–C films, i.e., 5×10^{-17} m^3/Nm. The COFs of DLC films against 100Cr6 steel show values of around 0.1.

3.2 Tribological characterisation of the thin Al foils sliding on the PVD- and CVD-coated hard-metal balls

Figure 1 shows the evolution of the COF of DLC (black line) and WC–C (grey line) coated hard-metal balls for different temperatures: RT, 70°C, 100°C, 125°C and 150°C; sliding on thin aluminium foil. The uncoated hard-metal balls exhibited values of the COFs of up to 1.1, decreasing progressively to 0.6 after 2,000 cycles [17]. The COF of WC–C-coated ball shows a non-continuous evolution, characterised by a low COF region of between 0.3 and 0.4 during the first 2,000 sliding cycles, after which the COF of the coated balls increases abruptly to 0.6–0.8, showing a high dispersion around the average values. In the case of the DLC-coated balls, the COF exhibited constant values of around 0.1 for all of the tests performed at room temperature: These values of the COF are characteristic for other DLC films, reported elsewhere [12].

Test performed at 70°C involving WC–C-coated balls show that the transition from low COF to high COF occurs earlier than when performed at RT. At 100°C in fact, the contact interface of WC–C-coated carbides on aluminium foil exhibits high values of the COF after just a few sliding cycles [17].

The interface between the DLC-coated hard metal and the aluminium foil exhibits different tribological properties. At 70°C, the sliding is characterised by a running in, transitory over the first 2,000 cycles, in which the COF oscillates from 0.7 and 0.1 in the form of up-down abrupt jumps. After 2,000 sliding cycles, the COF stabilises at around 0.1. A similar behaviour was observed during tests performed at 100°C and 125°C. At 150°C, the COF evolves from 0.8 to 0.6 during the first few sliding cycles, increasing to 0.8 after 1,500 sliding cycles and never exhibiting super-low friction properties [12].

The contact area of the tribological pairs under investigation was inspected optically for various sliding cycles and test temperatures, in order to gain insight into the contact phenomena occurring at their interfaces. Figure 2 shows the optical images of the DLC and WC–C-coated hard-metal balls after different sliding conditions on aluminium foil, as labelled. The contact areas of the DLC surfaces after sliding 10,000 cycles on aluminium at (a) RT, (c) 100°C and (e) 125°C do not show the presence of aluminium from the foil. The contact areas of DLC surfaces tested at 150°C (Fig. 2f) show evidence of aluminium transfer just after 200 sliding cycles. In the case of WC–C

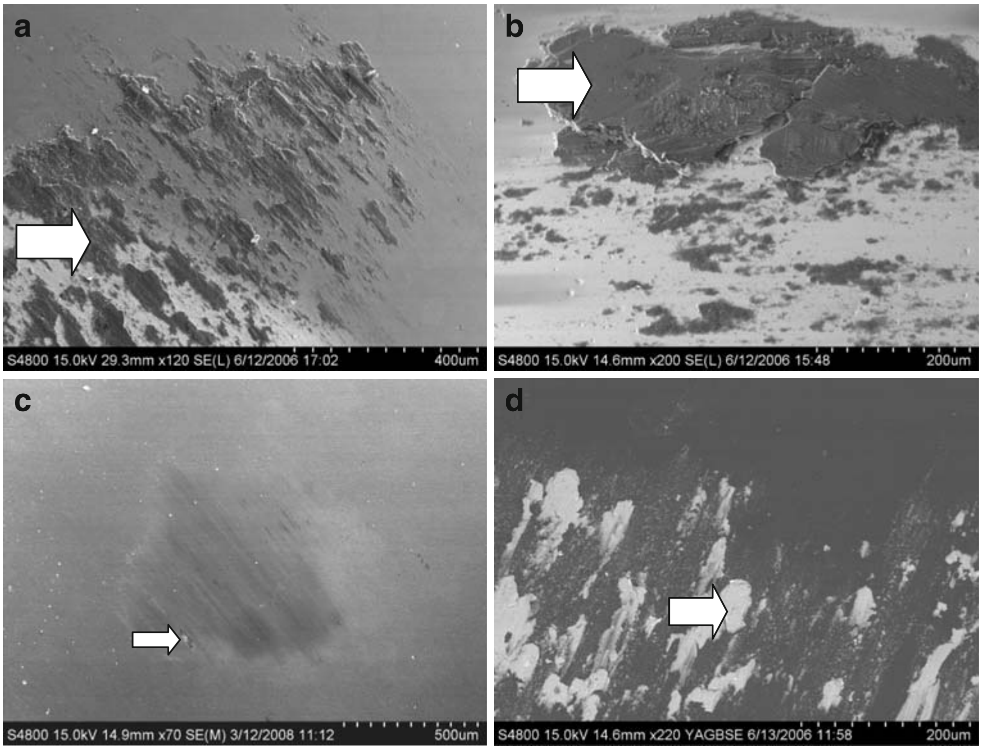

Fig. 3 SEM images of the contact areas of the hard-metal balls: **a** uncoated + 2,000 sliding cycles at RT, **b** WC–C coated + 1,500 cycles at 100°C, **c** DLC coated + 10,000 cycles at 100°C and **d** DLC coated + 200 cycles at 175°C after sliding on aluminium foil. The *arrows* point to aluminium clusters transferred onto the coated balls

films, the contact areas after (b) 3,000 cycles at RT and (d) after 1,000 cycles at 100°C already exhibit a massive transfer of aluminium.

A closer inspection of the coated hard-metal surfaces is presented in Fig. 3, which figure shows scanning electron microscope (SEM) images of the contact areas of the hard-metal balls for (a) uncoated after 2,000 sliding cycles at RT, (b) WC–C coated after 1,500 cycles at 100°C, (c) DLC coated after 10,000 cycles at 100°C and (d) DLC coated after 200 cycles at 175°C on aluminium foil. The arrows point to aluminium clusters transferred to the coated balls, according to elemental analysis by EDS. The uncoated hard-metal surfaces show transferred aluminium even after few sliding cycles performed at room temperature [17]. WC–C coated hard-metal surfaces also exhibit transfer of aluminium at temperatures around 100°C after few sliding cycles, as shown in Fig. 3b. In the case of the DLC surfaces, only small traces of aluminium were visible at temperatures below 125°C, as shown in Fig. 3c. Only those tests carried out at 150°C or higher showed a significant transfer of aluminium to the DLC surfaces (cf. Fig. 3d).

The inspection of the counter surfaces provides information on the effects of hard-metal surface degradation on the wear characteristics of the aluminium: This is better shown in Fig. 4, where the wear tracks produced on the foils by (a) WC–C coated after 1,500 cycles and (b) DLC coated after 10,000 cycles are depicted using SEM techniques after ball-on-disc tests at 100°C. Wear tracks produced by the WC–C-coated ball show grooves of nearly 1 mm depth. The inner part of the wear track exhibits thin strips along the sliding direction. These wear strips could be produced by the ploughing of hard aluminium-oxide micro-asperities formed on the hard-metal surfaces, or by other wear mechanisms.

In the case of the DLC-coated balls, the wear tracks were thinner than these produced by the WC–C-coated surfaces, even after a greater number of sliding cycles. Additionally, other zones exhibit groove widths similar to those produced by WC–C-coated surfaces. These zones may be affected by strong adhesive wear produced during a running-in sliding period, in which the COF of the DLC surfaces oscillated strongly. Elemental analysis did not show the presence of tungsten on the wear tracks of the aluminium foil, neither after sliding contact with WC–C- nor DLC-coated balls.

4 Discussion

The interfaces between different coated hard-metal balls and the aluminium foil have been compared for different contact temperatures after sliding under ball-on-disc experimental configurations. Magnetron-sputtered WC–C and PACVD-deposited DLC films showed different behaviour when sliding against the aluminium foil. At room temperature, both WC–C and DLC/aluminium contact interfaces exhibited COFs of below 0.2–0.3 during the first few sliding cycles. For a sufficiently great number of sliding cycles, the COF of WC–C surfaces increase due to aluminium transfer and the formation of an aluminium–aluminium interface, whereas the DLC surfaces exhibited low values of the COF even after 10,000 sliding cycles. In addition, there were significant temperature-provoked deviations between WC–C and DLC. For WC–C, the transition threshold from low-to-high COF diminished as the contact temperature set for the sliding test is increased. It was reported previously that this transition is triggered by the sticking of aluminium to the WC–C surface, forming an aluminium–aluminium tribo-surface, characterised by high COFs (0.5–0.7). In fact, the sticking phenomena of aluminium on WC–C surfaces were almost immediate at temperatures greater than 100°C.

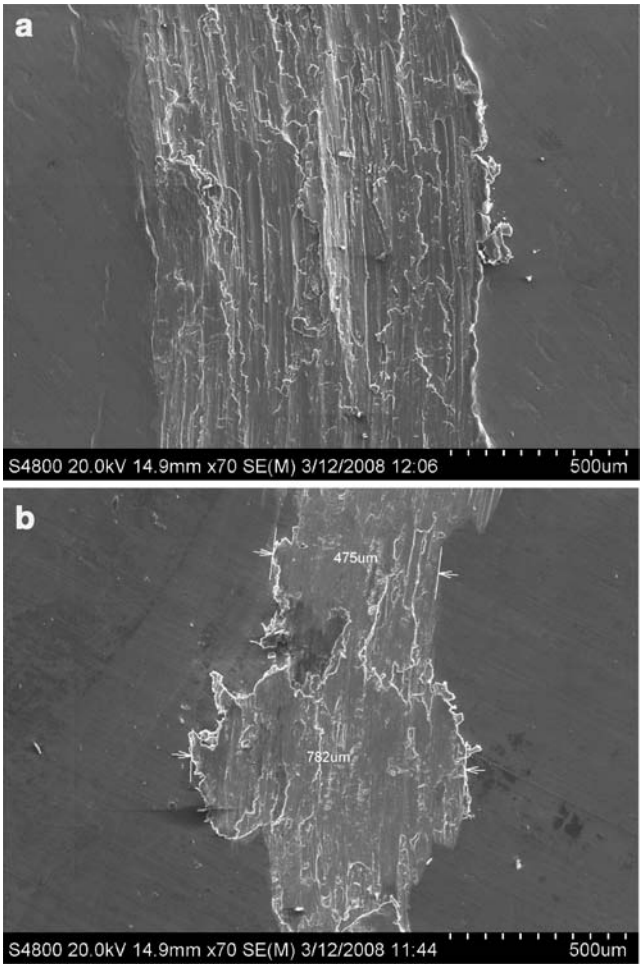

Fig. 4 Wear track produced at 100°C on aluminium foils: **a** WC–C coated after 1,500 cycles and **b** DLC coated after 10,000 cycles, as measured by SEM

PACVD-deposited DLC on aluminium surfaces exhibited running-in periods characterised by highly variable COFs. It is possible that these running-in periods are associated with the formation–destruction of aluminium–aluminium transitory interfaces. This feature was not confirmed from the data shown in this work, and further SEM analysis of such interfaces needs to be done to provide evidence of this feature. After the transitory running in, the COF of the DLC/aluminium interfaces drops to below 0.1 over not less than 10,000 sliding cycles, even at temperatures as high as 125°C. These results clearly indicate that the DLC films are more effective in preventing the aluminium from sticking on the hard-metal surfaces. At greater temperatures, the aluminium transfers easily to the DLC films, forming again aluminium–aluminium interfaces of high COFs.

5 Conclusions

Micro-manufacturing applications based on the severe plastic deformation of working materials are often limited due to galling, especially when the contact areas between the tool and the working material are in the sub-millimetre scale [1]. These features are well reported to produce severe problems during processing or demoulding. In this context, the utilisation of solid lubricious films showing low adhesive COFs and a high capacity to replicate the features of precision-tooling elements, e.g. dies or moulds, is strongly demanded. Since the PACVD DLC film is well reported to closely mimic the surfaces on which it is deposited [12] and given its good anti-galling properties against ductile metals such as aluminium, it is therefore postulated that this film is a feasible solution for particular micro-manufacturing applications, where friction effects need to be avoided without the use of oil–lubricants.

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