EMPIRICAL EVALUATION OF SPIN-ON-GLASS-LAYERS ON STEEL SURFACES BY WEAR TESTS[I]

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ABSTRACT

Dies and moulds with high precision surfaces are being used in various branches, e.g. in the plastic industry where the surface finish are conventionally performed by manual polishers. With ever increasing demands of shorter lead times and reduced costs, efforts have been made to automate this finishing process. This paper presents an empirical study performed to test durability properties of SOG (spin-on-glass)-layers on steel surfaces. The results showed that the thin coating last longer than the thicker ones, and that the harder coatings withstood wear significantly better than the steel reference samples thus motivating further investigations.

Keywords: Injection moulding, surface finish, tribometer, ball-on-disk, SOG-layers.

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INTRODUCTION

Surface finishes on dies and moulds for injection moulding are commonly manually polished into mirror like appearances, a process that are cumbersome, time-consuming and highly dependent on the individual polishers. Automated polishing techniques, e.g. various robot assisted polishing equipment [1-4], are therefore of great interest within industry. They are expected to increase the reliability of the production process by achieving consistent surface finishes from tool to tool, to reduce production time and costs, and to improve working environment for operators/polishers by minimizing the amount of vibrating hand tools and monotonic work positions/operations.

This paper considers a newly developed method for surface preparation of tools – coating of hydrogen silsesquioxane (HSQ). The mould surface is covered by a thin layer of liquid reactive silicon oxide precursor which cross-links during heat treatment at 450 °C and forms a hard, durable coating of amorphous silica. SOG-layered surfaces have been reported to reduce surface roughness' (peak-to-valley roughness) 10 times for paste polished steel samples [5], which should be comparable to surface finishes on polished tool steels. In situ tests have demonstrated that the coating was durable for more than 66.000 replications in injection moulding processes without loss of surface fidelity [6]. Previous research on friction tests of SOG-layered steel samples were not found by the authors. However, HSQ is used within other areas, e.g. in the semiconductor industry where its low dielectric constant, and excellent gap-fill and planarization properties are of advantage [7-8].

This work summarises initial experimental studies aiming at providing knowledge of the properties of the HSQ-coatings. Further studies, including more application oriented tests, are planned.

EXPERIMENTAL SET-UP

Five different types of surfaces were investigated (see Table 1-2); SOG-layered metal surfaces with three different layer thicknesses (substrate: ingot casted material ground and polished with 9 µm diamond paste), one ingot casted (IC) and one electro slag remelted (ESR) material. Both the IC and ESR samples were ground and manually polished to mirror-like finishes. The surface roughness', based on interferometric measurements, are represented by the Sq (Root-mean-square), St (Total height), and Str (Texture-aspect ratio) values, see Table 2. The hardness of the coating is considered to be at least twice as the one for the ESR steel.

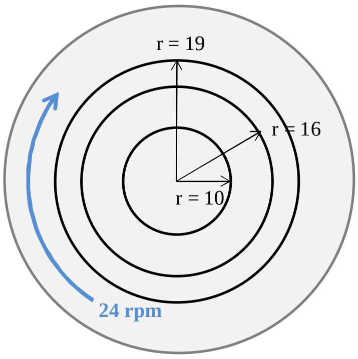
Table 1. Sample properties – layer thickness and hardness.

Sample	Layer thickness [µm]	Hardness [GPa]
A	0.9	-
B	1.3	-
C	1.7	-
IC	-	1.3
ESR	-	1.6

Table 2. Surface parameters based on interference measurements (measurement area 600x800 µm).

Sample	Sq [nm]	St [nm]	Str
A	45±5	770±30	0.7±0.1
B	30±5	470±30	0.7±0.1
C	30±5	430±30	0.7±0.1
IC	5±2	180±30	0.5±0.1
ESR	5±2	65±30	0.8±0.1

The samples were rubbed against a hard steel ball (440-C, HRC 62) of 6.35 mm radius on a commercial pin-on-disk tribometer [9]. A rotation speed of 24 rpm, a load of 8 N and a time of 3 minutes were chosen. The test was repeated three times on different radii, 10, 16 and 19 mm, which means that the ball passed each point in the wear traces equal times but at different linear velocities, see figure (Fig. 1). The electrical resistance signal was recorded as an indicator of coating fracture, the diameter of the worn area on the steel balls, i.e. the diameter of the contact area after 3 minutes testing, as a measure of ball wear.



Radii, r [mm]	Linear velocity, v [mm/min]
10	1500
16	2400
19	2850

Figure 1. Schematic view of a sample showing test radii and corresponding linear velocity.

Both the test-samples and ball surfaces were measured by a portable USB-microscope[1] without removing the test samples from the setup. After the tests, the samples were disassembled and measured by an optical interferometer[2] (quoted vertical resolution up to 0.1 nm, sampling 1 µm) to evaluate the size of the wear traces. A scanning electron microscope[3] (SEM) in the secondary electron mode was used to confirm the interferometer measurements. An elemental analysis of the traces was performed by the SEM combined with an energy dispersive x-ray spectrometer[4] (EDS).

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RESULTS

Layer thickness

Sample A with the thinnest coating was less damaged after 3 minutes testing, see figure (Fig. 2). The others were damaged to various levels; from continuous grooves to interrupted grooves. Based on the resistance curves, sample A (the trace at r = 10 mm) broke after some few seconds whilst sample C broke after ca. 70 seconds, see figure (Fig. 3). The arrow in figure (Fig. 3) indicates when the coating is considered to be fractured, i.e. when the steel ball is in contact with the underlying steel surface (the substrate).

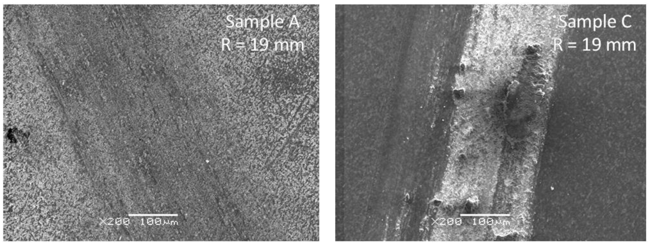

Figure 2. SEM images of all SOG-layered samples after 3 minutes testing, i.e. completed tests; left image shows sample A (r = 19), right image sample C.

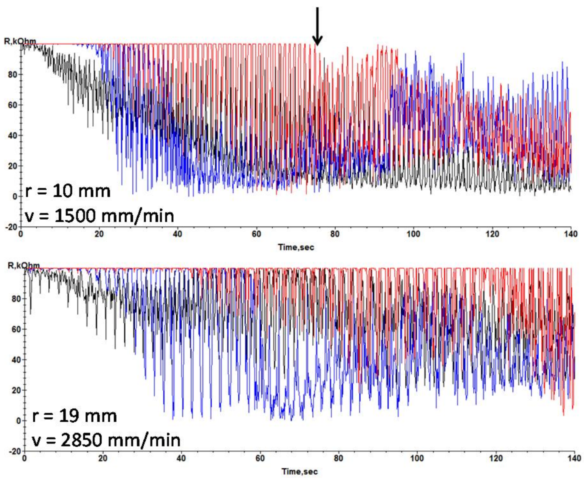

Figure 3. Electrical resistance versus time; the upper graph for r = 10 mm, the lower for r = 19 mm. Sample A is represented by a black line, sample B with a blue, and sample C with a red. The arrow marks when the layer on sample C is considered to be fractured.

Linear velocity

The resistance measurements indicated that time to fracture was longer for the highest linear velocity compared to the lowest one, see figure (Fig. 3) and (Fig. 4). The time to fracture was doubled for sample B, from 30 to 60 seconds, when the linear velocity was increased from 1500 mm/min to 2850 mm/min.

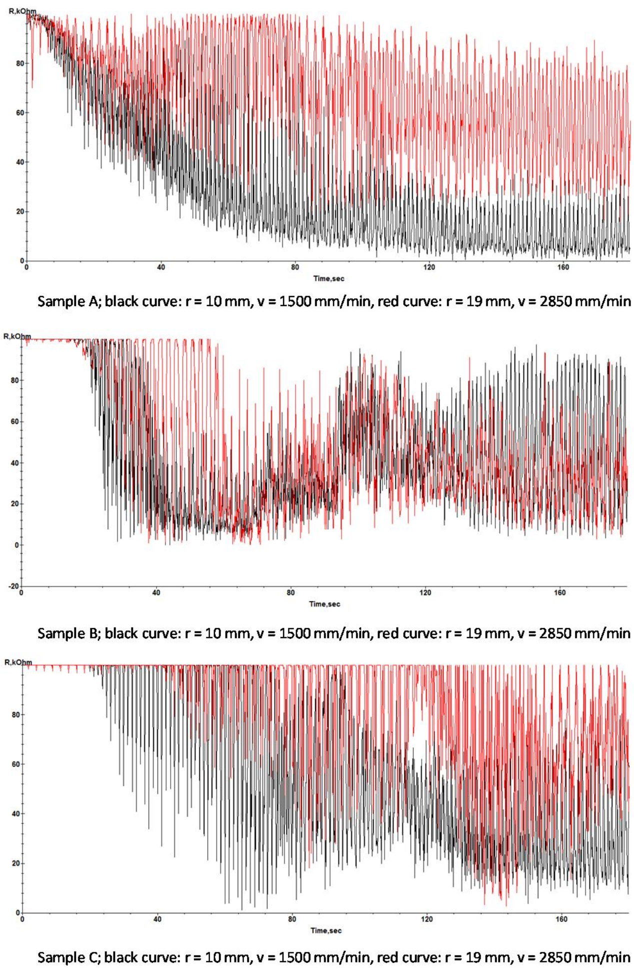

Figure 4. Electrical resistance versus time; the upper graph shows sample A for r = 10 mm (black curve) and r = 19 mm (red curve), the middle sample B, and the bottom sample C.

Wear

The SOG-layer seemed to be worn down by small stepwise torn off layer parts, see figure (Fig. 5), Sample A illustrating three wear levels – a slightly scratched surface, beginning of torn out layer parts, and a complete wear trace. The coating on Sample C is completely broken after three minutes, also the wear tracks were observed to be wider than those on Sample A.

The elemental analysis verified the broken coatings, and indicated that steel material was smeared onto the sample, as particles and smeared layers, see figure (Fig. 6). The coating seemed to be broken in front of these smears.

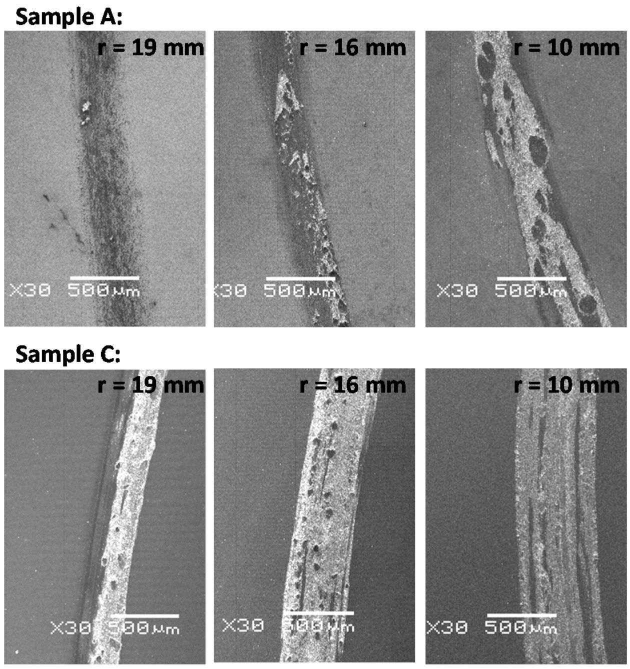

Figure 5. SEM images of sample A and C after 3 minutes testing at three different radii illustrating different wear levels; r = 19 mm is considered to be less worn, r = 10 mm the most. White area indicates fracture.

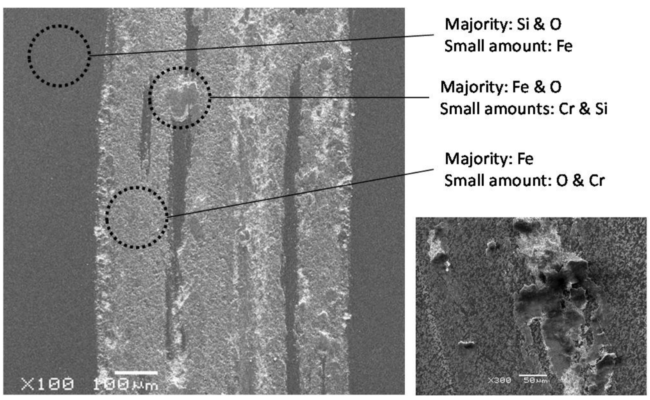

Figure 6. SEM image of a wear track on sample C; the small image show the smeared metal layer on sample B.

Coated and uncoated surfaces

Figure (Fig. 7) shows the different wear traces formed on sample A, C and IC, figure (Fig. 8) the width of the traces; the wear trace on the uncoated sample IC was deeper than the wear traces on sample A and C. The depth of the wear trace on sample A was hardly visible. Also the diameter of the wear mark on the steel balls indicated a higher wear rate for the coating-ball contact than for the metal-ball contact, see figure (Fig. 9).

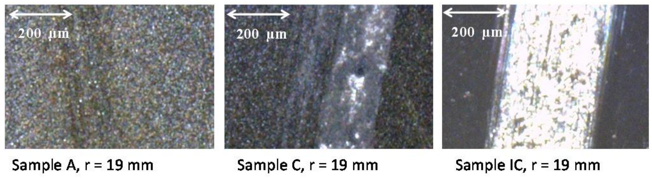

Figure 7. USB-microscope images of sample A, C and IC after 3 minutes testing at radii 19 mm illustrating the different wear traces formed on the samples.

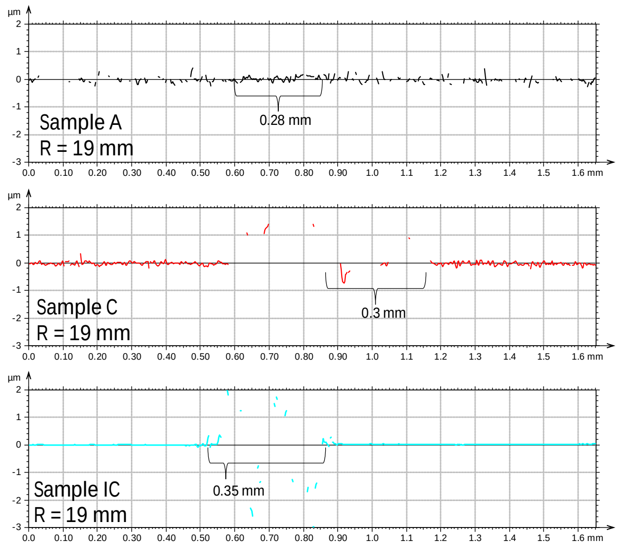

Figure 8. Cross sections of different wear traces on the HSQ-coated samples A and C, and the uncoated sample IC after 3 minutes testing. The profiles are based on interferometric measurements.

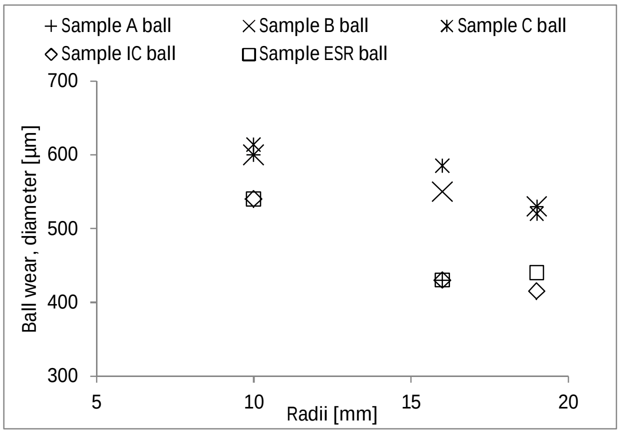

Figure 9. Ball wear, diameter vs. trace radii; the steel balls in contact with the uncoated steel samples were less worn compared to those in contact with the coated samples.

DISCUSSION

Surface topography

Sample B and C were measured to be smoother than sample A, which probably is due to the ability of thicker coatings to fill up surface irregularities better than thinner ones. The tool steel surfaces were clearly smoother, and more 'mirror-like' in their appearance which also is an important factor to take into account – mould users often link surface appearance to mould function and surface quality.

The lower level of surface damage on sample A could be linked to its surface topography since higher surface roughness should lead to smaller contact area, higher contact pressures and higher wear on the steel ball.

Layer thickness and linear velocity

The thinnest coating, sample A (r = 19 mm), was less damaged and only small parts were visibly fractured. However, clear wear traces were formed for the r = 16 and 19 mm tests. Thus, the thinnest coating seemed to be better for the higher velocity, and the thicker ones for the lower velocities, see figure (Fig. 2) and (Fig. 3).

Theories on ceramics, e.g. [10], mean that ceramics have two main wear regimes, a 'high' and a 'low' level. The 'low' level includes tribo-chemical wear or plastic deformation on the micro level, the 'high' level considers surface fragmentation. The transition between the two occur at a certain value – the PV-limit – which is equal to the contact pressure times the sliding velocity. This means that the contact pressure is the main factor in the beginning of the test when the contact area is small, i.e. the layers are fractured due to high contact pressures in the beginning of the tests.

Wear

The SOG-layer seemed to be worn down by small stepwise torn off layer parts, see figure (Fig. 5) illustrating three wear levels – a slightly scratched surface, beginning of torn off layer fragments, and a complete wear trace. The fluctuating resistance values in figure (Fig. 4) are explained by reoccurring contact and non-contact situation between the ball and the sample.

Coated and uncoated surfaces

The coating showed positive wear properties; by the simple conclusions in this study the wear was clearly reduced when the HSQ-coating was introduced (based on the size of wear traces). As expected, the wear rate of the steel balls was higher for the coating-ball contact than for the uncoated-ball contact due to the higher hardness of the coatings.

CONCLUSIONS

This empirical study initially show that

- sample A was less damaged, thus the thin SOG-layer withstood wear better than the thicker ones (sample B and C),
- the wear traces on the coated samples were wider and shallower than the wear traces on the steel samples,

- the wear on the balls were higher for those used on the coated surfaces compared to those used on the steel samples.

Complementary studies are needed to e.g. better understand the relation between layer thickness, contact pressure and linear velocity. Further, other methods/techniques, like scratch tests, should be used to better understand wear mechanisms, mechanical properties of SOG-layered surfaces and, their coverage and adhesion ability to underlying substrate.

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