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Wear investigation of selective α-Fe_2O_3 oxide layers generated on surfaces for dry sheet metal forming

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Abstract

To realize a dry sheet metal forming process, α-Fe_2O_3 oxide layers were investigated regarding their friction characteristics, wear behavior and surface energy depending on different surface qualities of the specimens examined. The oxide layers were generated in a new hybrid batch furnace. The layer generation on all specimens of the tool steel 1.2379 used was performed at a target temperature below the annealing temperature (≈ 510 °C). Friction coefficients were examined with plane strip drawing tests. Wear experiments with oxidized wear specimens with variable surface topographies were carried out up to several thousand strokes per surface condition.

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Keywords: Dry metal forming; Oxidization; Oxide layers; α-Fe_2O_3 layers; Sheet metal; Friction coefficient, Wear testing; Wear behavior; Surface quality

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1. Introduction

The metal forming industry is strongly interested in the control of the tribology in forming operations. In general, lubricants are employed to improve friction conditions and prevent forming tools from wear. Particularly from an ecological point of view, the utilization of mineral oil based forming lubricants is considered critically. Some of the additives (e.g. chlorine) are harmful and difficult to dispose in an environmentally friendly manner [1]. Furthermore, parts that are manufactured using forming oils have to be cleaned elaborately to prepare them for subsequent processes like joining or painting. This cleaning procedure often contains numerous cleaning stages that make the cleaning process itself expensive. Thus, technologies are needed which work ideally without lubricants.

The solution regarding many challenges of green tribology in forming industry is the realization of a dry forming process. A dry forming process is defined in [2] as "…a process where a workpiece leaves the forming tool without the necessity of cleaning or drying before further production steps such as coating or joining processes.". To achieve such a dry metal forming process different approaches in sheet [3, 4] as well as bulk metal forming [5] are studied in current research activities. Another opportunity for the realization of a dry sheet metal forming process is the selective oxidation of tool steel surfaces as shown in [6]. Selective oxide layers can be generated directly on the substrates surface as a result of a specific heat treatment in a well-defined atmosphere. Depending on the substrates alloy, different types of oxide layers after heat treatment are possible. In previous investigations promising results regarding good friction conditions and wear resistance of an α-Fe_2O_3 oxide layer on 1.2379 tool steel were obtained [7, 8]. In these studies, the surface topography was kept constantly at an arithmetical mean height of about $S_a \approx 1$ µm. To gain more information about the coherence between topography, layer adherence as well as friction and wear characteristics further investigations were carried out in the present study.

2. Selective oxidation

The reducibility of an oxide system for a given oxygen partial pressure and temperature is defined as a function of its free enthalpy of formation ΔfG. Hence, process conditions must be adjusted sensitively to generate a specific oxide layer system. For steels, the base metal can react to four different types of iron oxides, particularly hematite (α-Fe_2O_3), maghemite (γ-Fe_2O_3), magnetite (Fe_3O_4) and wüstite (FeO), [9]. As described in [8], especially α-Fe_2O_3 layer systems have been characterized for the process conditions of interest here.

In the present study, selective oxidation of the specimen surfaces was performed by heat treatment in a tubular furnace. Stationary conditions were achieved by controlling the composition of the atmosphere, the process temperature and the gas flow using Argon (Ar) with a purity of 99.996 vol.-% as inert shielding gas. The heat treatments were conducted convectively at a constant oxygen content of 0.03 vol.-% measured by a lambda probe. The experimental setup of this heat treatment method is described in detail in [10]. The specimens were heated up (5 °C/s) to 510 °C and hold isothermally for 60 minutes prior to cooling down to ambient temperature.

3. Materials and methods

All experiments were carried out with the sheet material DP600+Z (EU alloy grade 1.0936) with an elemental composition of 2.0% Mn, 1.5% Si, 1.0% Cr+Mo+Ni, 0.14% C, 0.07% P, min. 0.015% Al, 0.015% S, 0.005% B (in wt.-%). The nominal sheet thickness of the material before testing was $s_0 = 0.96$ mm. The surface of the sheet metal was hot-dip galvanized with an amount of 50 g/m² on each side of the material. The tool steel specimens used for all investigations were made from tool steel X153CrMoV12 (EU alloy grade 1.2379) with an elemental composition of (wt%) 12% Cr, 1.55% C, 0.9%V, 0.8%Mo and balance Fe hardened to 58 ± 2 HRC. The tool steel surfaces were examined in three conditions whereby the roughest surface was the topography after precision turning (target S_a-value 0.8 µm) of the specimens. The initial surface was finished by ground finishing (SiC 1000, target S_a-value 0.5 µm) and polishing (with 0.3 µm diamond suspension, target S_a-value 0.2 µm) in the hardened condition to generate the second and third surface condition.

3.1. Strip drawing and wetting tests

For the determination of topography depending friction coefficients, and thus the correlation between topography and resulting oxide layer, plane strip drawing tests were conducted. The experimental strip drawing tests were carried out on a universal test bench shown in [6]. Within the strip drawing tests for each specimen five sheet strips (length: 700 mm; width: 35 mm) were drawn over specimen surfaces, Fig. 1.

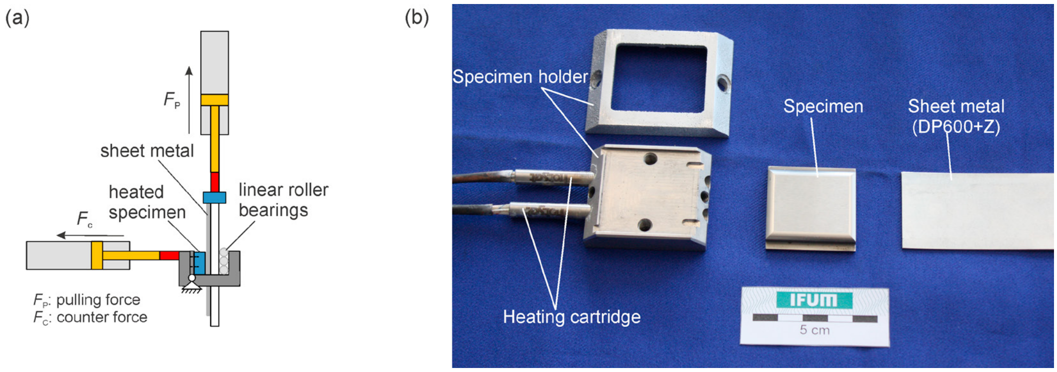

Fig. 1. (a) Principle of plane strip drawing test; (b) specimen geometry for investigation of friction characteristics.

To avoid edge effects in the contact area between the sheets edges and the specimen's surface, the specimen geometry shown in [6] was modified. Specifically, the sheet metal width was increased from 20 mm to 35 mm to realize a contact between sheet metal and specimen without edge effects on the surface. The strip drawing tests were carried out at a defined temperature of 100 °C while the specimens were heated up using a heatable specimen holder equipped with two heating cartridges (each 6.5 mm diameter, 40 mm length, 100 W power) including type J thermocouples. The heating of the specimens was employed in order to mimic a stationary forming process. In real forming operations, especially in high volume production, tools are heated due to the dissipation of inner (material) and outer (between surfaces) friction. The investigations were carried out with a surface pressure of 10 MPa and a velocity of 20 mm/s. The plane specimens with the three selected surface conditions are shown in Fig. 2. The glance shows the difference in the surface quality whereby the lens of the light microscope is reflecting for the polished condition.

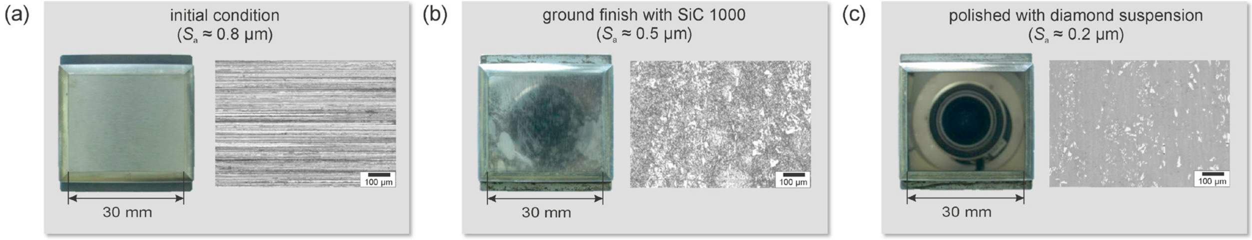

Fig. 2. (a) Oxidized specimen for plane strip drawing tests in initial condition after manufacturing; (b) oxidized specimen for plane strip drawing tests ground with SiC 1000 abrasive; (c) oxidized specimen for plane strip drawing tests polished with diamond suspension.

For the characterization of the wettability of the tool steel surfaces with the investigated surface conditions, samples with a diameter of 16 mm and a height of 4 mm were prepared. For every condition, a drop of distilled water was applied to the surface, while the contact angle was determined optically (light microscope). This method describes the wettability as a function of surface energy for a constant surface tension of the chosen fluid (water) to quantify the influence of different surface conditions on selective oxidation effects.

3.2. Wear investigations

The wear tests were carried out on a wear test bench as presented in [8]. The detailed investigation of the very thin selective oxide layers is challenging. Therefore, a two-part specimen with a small cylindrical active part (radius $r = 8$ mm, length $l = 50$ mm) and a heatable socket was developed and used for the wear investigations [7, 8]. The test bench including the assembled two-part specimen and a schematic is included in Fig. 3.

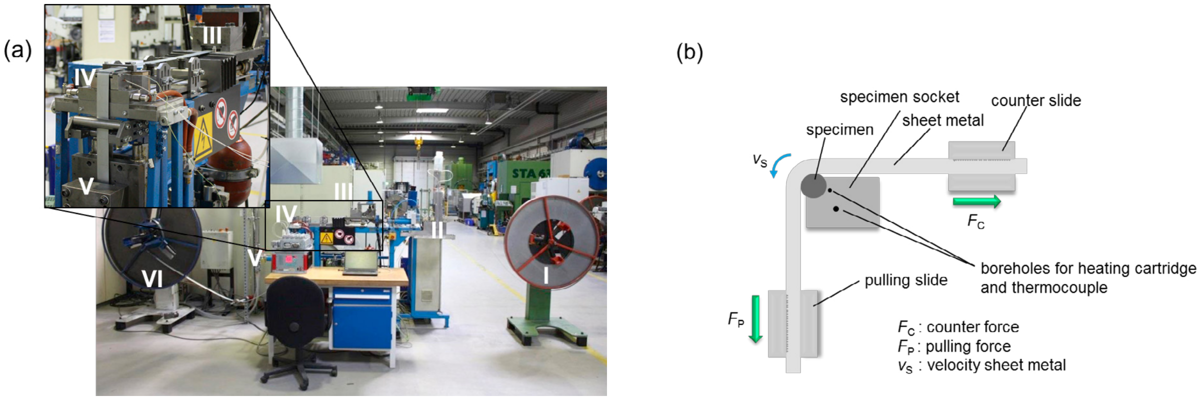

Fig. 3. (a) Sheet metal wear test bench with I: decoiler, II: cleaning station, III: counter slide, IV: specimen, V: pulling slide, VI: coiler (left picture); (b) principle of the wear test bench (right picture).

In this setup, the force of the test bench is build up by hydraulic pressure. The velocity v as well as the surface pressure and the path of stroke can be adjusted by the operator of the machine. On the test bench the sheet metal is drawn over a 90° radius. This testing situation was chosen to mimic a deep drawing process at the drawing edge, which represents an area with high load. For the investigations a path of 60 mm per stroke, an average surface pressure of about $\bar{p} = 95$ N/mm² and a temperature of 100 °C were used. Even if the contact pressure in deep drawing of DP600 is higher in real forming operations for the basic investigations in this study a moderate contact pressure was chosen, cf. [8]. The determination of the surface pressure was carried out by the use of pressure indicating films. Each stroke of load took about $t = 1.5$ s at about $v = 40$ mm/s. To ensure a dry forming operation the material initially passed a multiple step cleaning station (10% Tickopur R33 cleaning solution) where the prelube oil was removed. In addition to the cleaning station, the sheet metal was cleaned with an ethanol (> 96%) soaked sponge prior to getting in contact with the tool steel specimen to avoid residues from the cleaning solution. Subsequently the material was drawn over the specimen with a drawing radius of $r = 8$ mm. The drawing of the sheet metal was realized by the use of two slides whereby each was mounted on a linear slide. While the operator was adjusting the counter force F_C, the pulling force F_P is a resulting force, which is about 20% higher than F_C. For the wear investigations specimens with the described three surface conditions – each condition oxidized and not oxidized as references – were tested with 500 and 2000 strokes of load. The experimental matrix of the whole wear study is shown in Table 1.

Table 1. Experimental matrix of wear investigations.

Specimen	Surface layer	Surface condition	Number of strokes
A1	α-Fe_2O_3	initial	500
A2	α-Fe_2O_3	initial	2000
RA1	-	initial	500
RA2	-	initial	2000
B1	α-Fe_2O_3	ground finish	500
B2	α-Fe_2O_3	ground finish	2000
RB1	-	ground finish	500

RB2	-	ground finish	2000
C1	α-Fe_2O_3	polished	500
C2	α-Fe_2O_3	polished	2000
RC1	-	polished	500
RC2	-	polished	2000

3.3. Surface analysis

The surfaces of all specimens were analyzed using various optical methods. Each measurement was carried out at an angle of 45° to the drawing direction. At this angle, drive in and out effects of the sheet metal are nearly negligible. A digital light microscope, Keyence VHX-1000, was applied for imaging the surfaces at several magnifications. To quantify the changes of the specimens topography a confocal laserscanning microscope, Keyence VK-9710, was used. In addition, a scanning electron microscope, Zeiss Supra 55 VP, which was equipped with several detectors, was used for high resolution imaging. Secondary electron and inlens detectors were employed within the present study.

4. Results and Discussion

The first results that will be discussed are those of the plane strip drawing tests for the experimental determination of friction coefficients with variable topographies shown in Fig. 4.

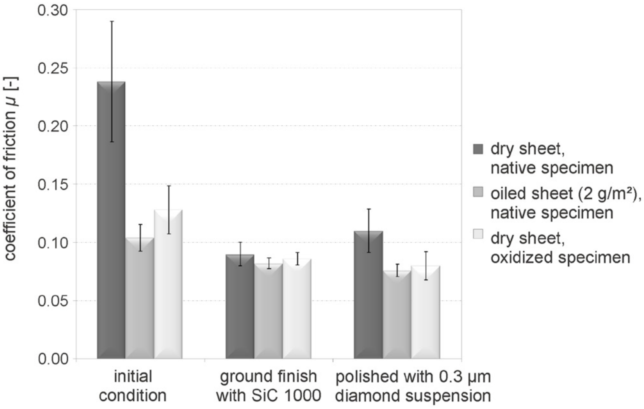

Fig. 4. Results of plane strip drawing tests with variable topography conditions; experiments carried out with surface temperature of 100 °C and an average surface pressure of 10 N/mm².

The data show a significant difference in friction coefficient between the topographies as well as the lubrication systems. For each topography condition, the dry references show the highest average friction coefficients as well as highest scatter. The lowest average coefficient of friction for each condition was determined for the tribosystem consisting of a native specimen and oiled sheets (lubricated with 2 g/m², deep drawing oil WISURA AK3080). Still, the tribosystem with oxidized specimens and dry sheets shows coefficients of friction similar to the lubricated tribosystem. Furthermore it can be seen, that the scatter for the oxidized specimens is of similar magnitude as for lubricated system. It is assumed that the reason for this is the separation layer between the steel surfaces. On the one hand, the oil is separating the steel surfaces and on the other hand, the oxide layer is acting as a separation layer that avoids the unfavorable direct steel on steel contact. Contrary to the assumption that the polished surfaces are resulting in the lowest coefficients of friction, the ground finished surfaces are showing similar results to the polished ones. The average friction coefficient of the dry sheet with native specimen is even higher for the polished surface. These results might be attributed to the true contact area which is enlarged at finer surfaces. The reduction of protruding peaks on the surface seems to have a significant influence on the sliding resistance, and thus, the friction characteristics. Based on these results a ground finishing of oxidized forming tool surfaces seem to be sufficient regarding the friction characteristics, which is also beneficial from an economic point of view compared to polishing. After the determination of friction coefficients wear investigations with identically prepared wear specimens were carried out. SEM micrographs of the conditioned tool steel surfaces were taken prior to (virgin specimens) and after wear investigations (2000 strokes). In Fig. 5 (left side) the optical difference between oxidized and not oxidized specimens is shown exemplary in the polished condition before wear tests (a) reference not oxidized and (b) oxidized. On the right side in Fig. 5 the surfaces of oxidized specimens in each surface condition (initial (c), ground finished (d) and polished (e)) after wear tests are shown in various magnifications.

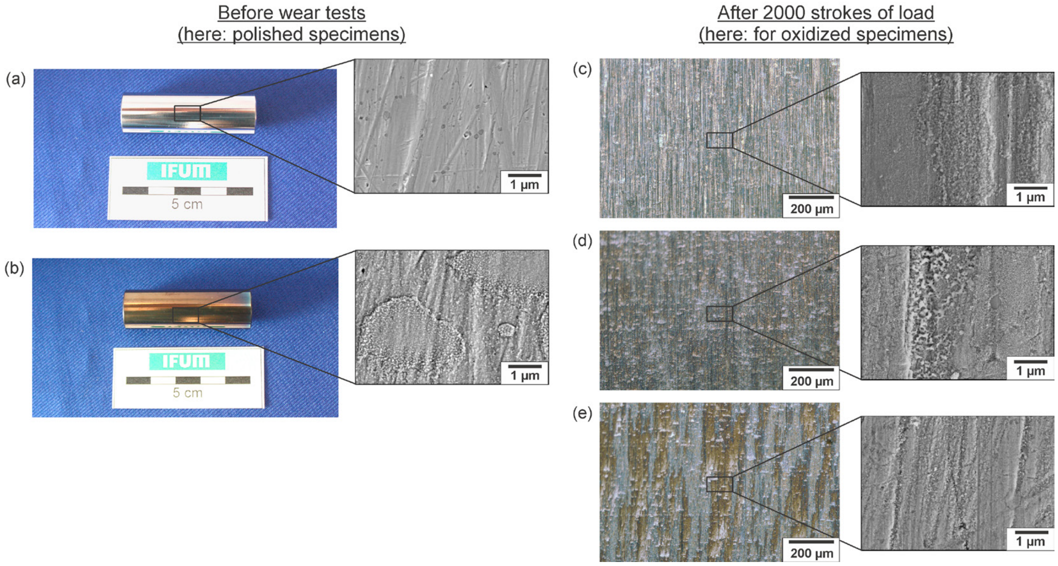

Fig. 5. (a) Reference diamond suspension polished wear specimen before wear (left) and SEM image (right), (b) oxidized diamond suspension polished wear specimen before wear (left) and SEM image (right), (c) light optical image of the surface of an oxidized specimen in initial condition after wear tests (left) and SEM image (right); (d) light optical image of the surface of an oxidized ground finished specimen after wear tests (left) and SEM image (right); (e) surface of oxidized diamond suspension polished specimen: light optical image (left) and SEM image (right).

The polished surface (Fig. 5(a)) features fine grinding grooves, which are also visible for polished oxidized untested (cf. Fig. 5(b)) and polished oxidized tested (cf. Fig. 5(e)) conditions. For condition (b) (polished oxidized) an oxide layer consisting of small oxide nodules on the surface appears, while chromium carbides occur at areas, which have not been covered by the oxide layer. Those precipitations seem to have been flattened during the wear tests, while oxide particles are still visible at the tool steel surface but to a lesser extent as prior to wear. For initial oxidized (cf. Fig. 5(c)) and oxidized ground (cf. Fig. 5(d)) conditions, oxide particles are also present, while the structure of oxide nodules has coarsened in contrast to oxidized polished surfaces.

As part of the wetting tests the contact angle θ, formed by the intersection of the liquid-solid interface and the liquid-vapor interface was determined. As summarized in Table 2, the contact angle θ does vary with the finishing procedure of the specimen surfaces. Moreover, finished specimen surfaces featured contact angles (< 80°) with higher wetting potential than the initial surface conditions, which achieve wetting angels of nearly 85°. In general, the surface conditions are favorable by featuring wetting angles lower than 90°, however, wetting angles are still quite high.

Table 2. Contact angles following different surface conditions.

Surface condition	**initial** (**$S_a \approx 0.8$ µm**)	**grinded** (**$S_a \approx 0.5$ µm**)	**polished** (**$S_a \approx 0.2$ µm**)
Contact angle Θ	83.9° ± 1.18°	80.1° ± 5.05°	73.3° ± 2.34°

To quantify the surface changes and gain knowledge about the wear effects, all examined specimens were analyzed by confocal laser microscopy before and after wear tests. According to [8] the S_a-value (arithmetical mean height) and the V_{vv}-value (dale void volume) were studied in detail, Figs. 6 and 7. On each specimen three areas with a measuring field size of 15 mm² were measured in 45° as described in 3.3.

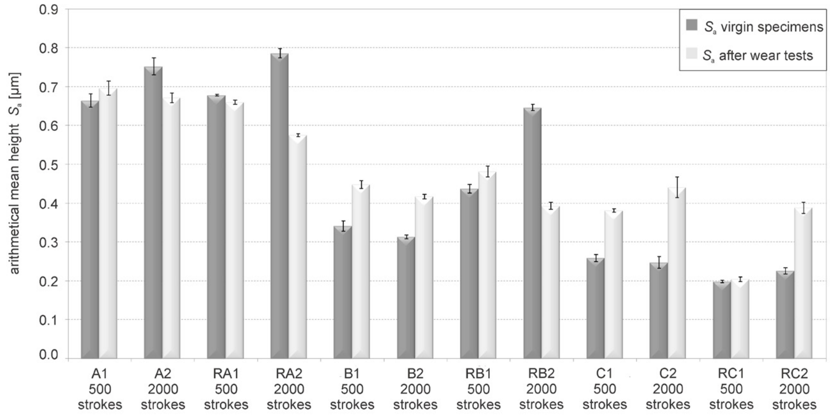

Fig. 6. Arithmetical mean height (S_a) of all investigated surfaces in 45 ° before and after wear tests.

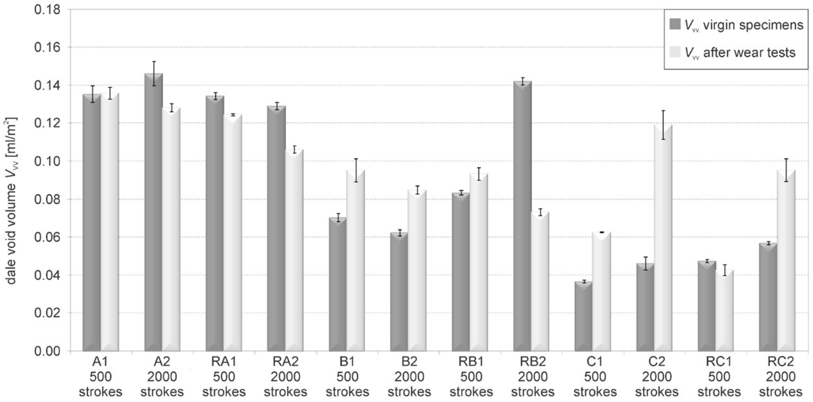

Fig. 7. Dale void volume (V_{vv}) of all investigated surfaces in 45 ° before and after wear tests.

The S_a-value shows a strong influence of the machining before the wear tests as well as an influence inside each test series between the oxidized specimens and the references. In most of the cases after 500 strokes of load, the change of the S_a-value is only minor. The experiments with 2000 strokes of load show a higher shift of the arithmetical mean height. Noticeable is that in test series A (initial surface) the S_a-value is decreasing after 2000 strokes while this trend is inverse (except for RB2) for the test series B (ground finished) and C (polished with diamond suspension). In general the S_a-value of test series C is not much lower than in test series B. This result can be attributed to the specimen's preparation. Both finishing methods were carried out directly after the manufacturing (initial condition). Thus between polishing and manufacturing no additional grinding operations were carried out. However, polishing with diamond suspension is the more complex method for finishing forming tools. A comparison between the oxidized specimen A2 and the appropriated reference RA2 shows that the S_A-value of the reference is decreasing stronger. This might be a stronger wear of the surface peaks on the one hand and higher zinc pick up as shown in [8] on the other hand. The test series B shows two different tendencies after 2000 strokes. While the S_a-value is increasing for B2 it is decreasing for RB2 after wear tests. Conspicuous for RB2 is that the S_a-value for the virgin specimen is much higher compared to RB1 machined the same way. It seems that the surface of RB2 exhibits more peeks even after ground finishing. A look on test series C shows a clearer trend. Both S_a-values (C2 and RC2) are increasing following the wear tests. The mean value of C2 is slightly higher but approximately within the scatter of RC2. Very similar observations were found for the parameter dale void volume (V_{vv}) that describes the volume of empty valleys in a surface as shown in Fig. 7. This parameter is useful to gain knowledge about debris (zinc) pick up in empty volumes or surface changes before and after oxidation. The tendencies of the dale void volume before and after the wear tests are equal to the trends of the arithmetical mean height. An increasing dale void volume for the ground finished and the polished surface could result due to micro grooves on the surface or zinc pick up. In summary, the results of the surface analysis indicate that finer surfaces result in an increase of the values (S_a and V_{vv}) during the wear tests. This seems to be a result of sliding wear as a consequence of a higher true contact area which is in accordance with the results of friction coefficients. Nevertheless, the oxide layer acts as a separation layer and avoids the tribologically unfavorable steel on steel contact. Even after 2000 strokes on each tested surface condition a remaining α-Fe_2O_3 layer was detected.

5. Conclusions

The conclusions of the study can be summarized as following:

- α-Fe_2O_3 oxide layers reduce the friction between tool and sheet metal compared to dry reference experiments. The friction characteristics of the oxidized specimens in all investigated surface conditions are comparable to the oiled tribological systems.
- Finer surfaces result in lower friction coefficients whereby no significant difference between ground finished surface and with diamond suspension polished surface was determined.
- For all investigated surface conditions, the oxide layer remained on the surface after 2000 strokes of load (≈ 120 m).
- Wear specimens with finished surfaces show tendencies to higher S_a- and V_{vv}-values after wear tests. These results seem to be attributable to a general roughening of the fine surfaces due to micro grinding combined with zinc pick up from the galvanized sheet metal used.
- For a dry sheet metal forming process with oxidized surfaces, a fine surface finishing of the tools regarding friction and wear characteristics seems to be unnecessary.

Acknowledgements

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