



Wear Testing of Thermally Oxidised Tool Steel Specimens with $\alpha\text{-Fe}_2\text{O}_3$ Layers

Deniz Yilkiran^{*1}, Daniel Wulff², Fahrettin Özkaya¹, Sven Hübner¹, Ulrich Holländer², Hans Jürgen Maier², Bernd-Arno Behrens¹

¹Institut für Umformtechnik und Umformmaschinen, Leibniz Universität Hannover, An der Universität 2, 30823 Garbsen, Germany

²Institut für Werkstoffkunde, Leibniz Universität Hannover, An der Universität 2, 30823 Garbsen, Germany

Abstract

The paper presents results of wear investigations of thermally oxidised tool steel specimens with an $\alpha\text{-Fe}_2\text{O}_3$ coating. The friction reducing ability of $\alpha\text{-Fe}_2\text{O}_3$ layers on tool steel was shown in previous studies. Reduced friction and the layer's stability are prerequisites to realise a dry sheet metal forming process that avoids the use of additional drawing oils. This in turn will make the forming process more eco-friendly.

The wear investigations were carried out on a wear test bench with the high strength sheet metal DP600+Z at a surface pressure of 80 MPa. Several thermally oxidised specimens were tested with different numbers of strokes in order to characterise the wear behaviour of the oxide layers. The surface analyses of the specimens were carried out using digital and scanning electron microscopy. Even after 5000 strokes (300 m of sheet metal) the 0.2 μm thin $\alpha\text{-Fe}_2\text{O}_3$ coating was only partly degraded so that a wear reducing effect can be exploited in actual applications.

Keywords: wear tests, $\alpha\text{-Fe}_2\text{O}_3$ layer, surface analyses

1 Introduction

The use of forming tools with a selectively oxidised surface is one promising approach for the realisation of dry sheet metal forming processes, where additional drawing oils are no longer necessary. In earlier studies [1, 2, 3] it was shown that thermally generated oxide layers can reduce the friction coefficient between the tool surface and a zinc coated DP 600 sheet metal. The oxide layer is acting as a low friction separation layer in this case. Depending on the heat treatment conditions and the process atmosphere in particular, different kinds of oxide layers form on the specimen's surface. Investigations of friction characteristics with two setups (with and without deflection) have demonstrated that $\alpha\text{-Fe}_2\text{O}_3$ layers on 1.2379 tool steel surfaces have the ability to reduce the friction coefficient between the tool steel surface and the DP600+Z sheet metal used [1, 3]. The investigations were carried out with unoiled sheet metals, cleaned from prelubes by using a 10 % Tickopur R33 cleaning solution. Reference experiments were done by using non-oxidised tool steel specimens with

prelube free sheet metals and non-oxidised specimens with oiled sheet metals (2 g/m² of the deep drawing oil Wisura AK 3080). The experiments showed that the friction coefficients of the oxidised specimens were significantly better than friction coefficients of non-oxidised reference specimens. Moreover, the friction coefficients of the $\alpha\text{-Fe}_2\text{O}_3$ layers were similar to the coefficients of oiled uncoated tool steel surfaces.

The positive effects of Fe_2O_3 regarding friction and wear reduction were also reported in earlier studies [4, 5, 6]. In [4], Fe_2O_3 particles with different diameters (30 nm, 300 nm, 500 nm and 1 μm) were systematically tested by pin on disk tests. The particles were placed on a steel disk and were tested with a steel pin. The friction reducing oxide layer was formed on the surface by sintering processes. After a run in period, the friction and wear decreased for all particle sizes used except for the 1 μm case to values smaller than without Fe_2O_3 particles.

In order to better understand the long term wear behaviour of the $\alpha\text{-Fe}_2\text{O}_3$ layer, further investigations were

necessary, and the objective of the present study was to address this issue.

2 Experimental setup

2.1 Wear investigations

The experimental investigations were carried out on a wear test bench by drawing fresh unused material over the specimen's surface with every stroke. This method ensured conditions similar to a forming process in actual production where also fresh material is used for each stroke. A dedicated multiple part wear specimen geometry (Fig. 1) was used for the investigations. For details see Ref. [7].

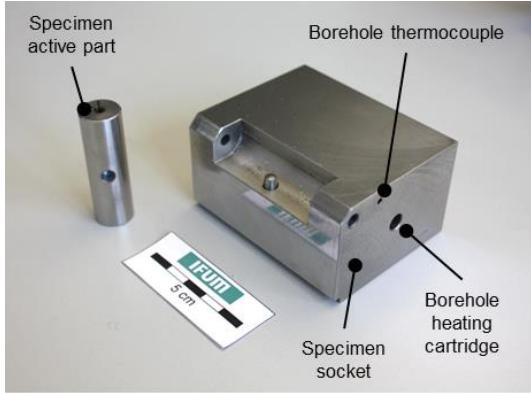


Fig. 1: Wear specimen including an active element (left) and a passive element (right)

The geometry shown in Fig. 1 was developed to ease non-destructive analysis (small active part), provide for economical benefits (separation of active and passive element) and allow for heating (with heating cartridges) and monitoring (with thermocouples) the temperature. By heating the 1.2379 tool steel specimens to $80^{\circ}\text{C} \pm 5^{\circ}\text{C}$ it was possible to quickly obtain stationary forming process conditions during the wear investigations. The temperature of about 80°C is also typical for commercial forming process with the high strength sheet material (DP600+Z in 0.96 mm) once stationary conditions have been reached. The resulting surface pressure between the specimen and the sheet



Fig. 2: left picture: Wear test bench with relevant units; a) decoiler, b) cleaning station, c) counter slide, d) specimen, e) pulling slide, f) coiler; right picture: principle of wear test bench

metal was determined by using pressure indicating films (Fujifilm HS - High Pressure Prescale) as reported in [6]. The wear specimen was mounted on the wear test bench shown in Fig. 2. The test bench is supplied with material from a coil. Fresh material is provided with each stroke from the decoiler (a). After decoiling, the material was cleaned from prelube oil (b). The drawing of the material over the drawing radius of the specimen (d) is realised on the test bench by using a counter (c) and a pulling slide (e). The counter force F_{counter} can be adjusted by the operator while the pulling force F_{pulling} is a resulting force which has to be higher than the adjusted counter force. In this way, the material is drawn over the specimens and finally fed to the coiler (f). During the forming process the forces as well as the displacements of the counter and pulling slide were recorded. For measuring the forces, a miniature load cell (HBM U9C) was used. The U9C load cell can be used for the measurement of tensile as well as compressive forces, which makes the set-up robust against backstrokes at the end of each cycle. The displacements of the counter and pulling slide were measured by using miniature wire sensors (Micro-Epsilon WPS-500-MK30).

For the present study, the material was cleaned from prelubes before forming (cf. Fig. 3). The cleaning station used purges the material in four stages from both sides:

- 1) dry sponge cleaning
- 2) wet sponge cleaning with 10 % Tickopur R33 cleaning solution
- 3) second dry cleaning
- 4) drying with compressed air (assembled on the test bench).

The macroscopic contaminations present on the sheet metal before and after cleaning were examined with a wipe test as shown in Fig. 3.

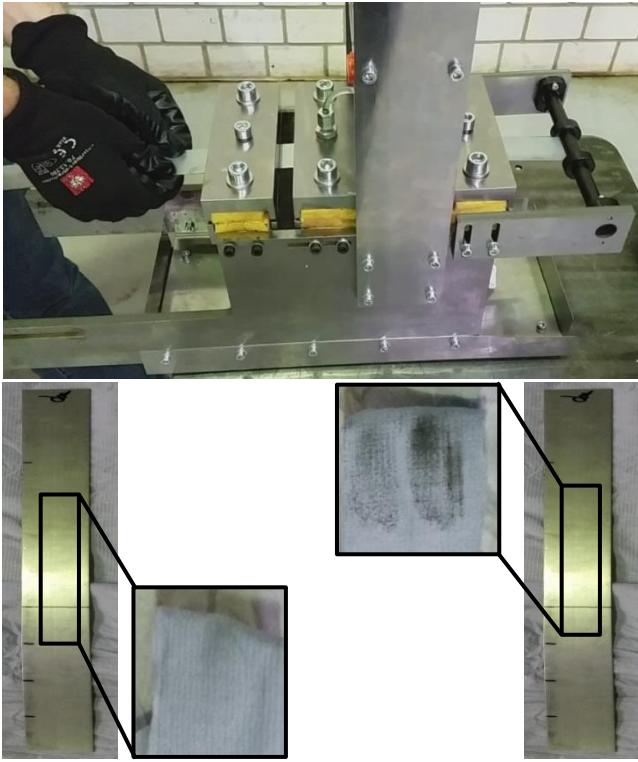


Fig. 3: Cleaning station without air nozzles (upper picture); wipe test before cleaning (bottom right picture); wipe test after cleaning (bottom left picture)

The wipe test, depicted in Fig. 3, shows the presence of significant macroscopic contamination of the sheet metal before cleaning. This can be attributed to the prelube oils which are used in commercial sheet metal production for corrosion protection. The wipe test in the left bottom picture shows a macroscopically clean sheet metal surface after passing the cleaning station within the first three cleaning steps. During the investigations the fresh material was continuously cleaned in the cleaning station. The cleanliness of the sheet metal surface was regularly manually checked with wipe tests. In case of detectable contaminations, the sponges of the station were cleaned or if necessary renewed.

In the present study two specimens with $\alpha\text{-Fe}_2\text{O}_3$ layer were tested. Table 1 shows the parameters of the wear tests. The specimens A1 and A2 were tested with equal surface pressures and process temperatures. While specimen A1 was tested up to 100 strokes (6 m) specimen A2 was loaded with 5000 strokes (300 m). The sliding speed in both cases was 40 mm/s.

Tab. 1: Test parameters of wear investigations.

Specimen	Strokes	Path per stroke [mm]	Surface Pressure \bar{p} [N/mm ²]	Temperature [°C]
A1	100	60	80	80 ± 5
A2	5000	60	80	80 ± 5

3 Results

Before and after the experiments on the test bench macroscopic and microscopic analyses were carried out. For this purpose the specimens were marked on the front face at three angles over the contact area which equals one quarter circle of the cylinder. These are the inlet area (0° angle), the central area (45° angle) and the outlet area of the sheet metal (90° angle). The inlet area showed the most distinctive layer wear, which was attributed to the bending of the sheet metal in this area. By contrast the outlet area had the lowest layer wear, and was partly uneven. Therefore the central area at an angle of 45° to the drawing direction was chosen as the area best suited for the analyses. Here, a uniform layer wear over the whole contact area between the sheet metal and the specimen's surface took place. All following data refer to this area. The observation of the specimen started with macroscopic screening followed by digital light microscopy and scanning electron microscopy (SEM). The light microscopy images were carried out using a digital light microscope (Keyence VHX-1000). For high resolution scanning an electron microscope (SEM), Zeiss Supra 55 VP, equipped with an energy dispersive X-ray spectroscopy (EDX) detector was employed. A secondary electron detector and a low acceleration voltage of 7 kV were used to achieve high resolution without significant charging effects of the oxide covered surfaces.

In Fig. 4 an untested specimen is shown at three magnifications. The top image in Fig. 4 shows an oxidised specimen with $\alpha\text{-Fe}_2\text{O}_3$ layer prior to testing. The whole surface of the specimen has a red/blue discolouration as a visible result of the surface oxidisation [1]. A higher magnification with a light microscope (middle picture in Fig. 4) reveals that not all of the surface is covered with the oxide layer. The highest magnification in Fig. 4 (bottom image) reveals the microstructure of the oxide layer. The $\alpha\text{-Fe}_2\text{O}_3$ layer looks like an arrangement of long polymer chains. The layer is covering the largest part of the section shown, but there are also other uncoated areas.

The coverage of the surface with $\alpha\text{-Fe}_2\text{O}_3$ strongly depends on the local composition of the substrate. The investigated hardened tool steel (1.2379) has a high chromium content of approx. 12 wt.-% and chromium rich precipitations with different sizes could be detected by SEM-EDX. Surface areas that featured precipitations were usually not covered by the $\alpha\text{-Fe}_2\text{O}_3$ unless these were very small. Thus, it can be reasoned that the metallic sparkling areas in the top image of Fig. 4 are uncoated chromium rich areas.

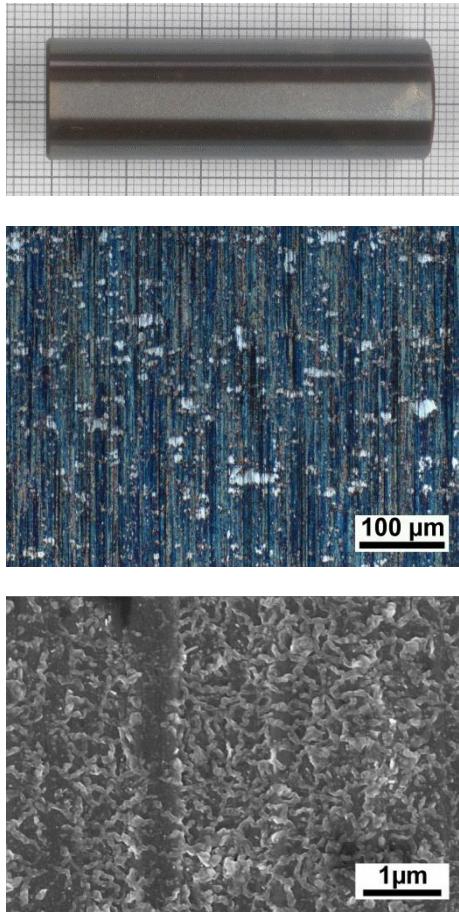


Fig. 4: Selectively oxidised $\alpha\text{-Fe}_2\text{O}_3$ specimen. Top: photo of untested specimen; mid position: digital light microscopic image; bottom: SEM image

Depending on the number of test cycles the surface of the specimens changed after the wear tests. In Fig. 5 the tested specimen A1 is shown after 100 strokes. The photo (top picture) in Fig. 5 presents the macroscopic appearance of the specimen after testing. While in the bending area of the sheet metal (0° angle) a layer thinning can be seen, the evaluated area (45° angle) shows no significant changes on the surface on the macroscopic scale. In addition, slight marks on the left and right side in the contact area with the sheet metal can be seen in the photo in Fig. 5. These are results of the sheet metal burr cutting into the surface. This phenomenon is increasing with the number of strokes but not relevant in the present context because in commercial deep drawing operations such contact conditions are not present.

The light microscopic image of specimen A1 (middle image Fig. 5) only shows slight changes as compared to the light microscopic image of the untested specimen (Fig. 4). Only a few additional scratches along the sliding direction (vertical in the image) can be seen. Furthermore metallic sparkling areas are now visible and it seems that the size of these areas has also increased. It can be assumed that their higher fraction is a result of a partial removal of the $\alpha\text{-Fe}_2\text{O}_3$ structure. The $\alpha\text{-Fe}_2\text{O}_3$ also covered the edges of chromium rich precipitations. This leads to the conclusion that the increased fraction of the oxide-free areas can be reasoned by the reduced bonding of the oxide to the substrate at these chromium rich areas. In the SEM image in Fig. 5 (bottom picture)

the surface appears very different, as compared to the SEM image of the untested specimen (Fig. 4). The structure of the oxide layer seems to be smoothed and rounded but is still visible.

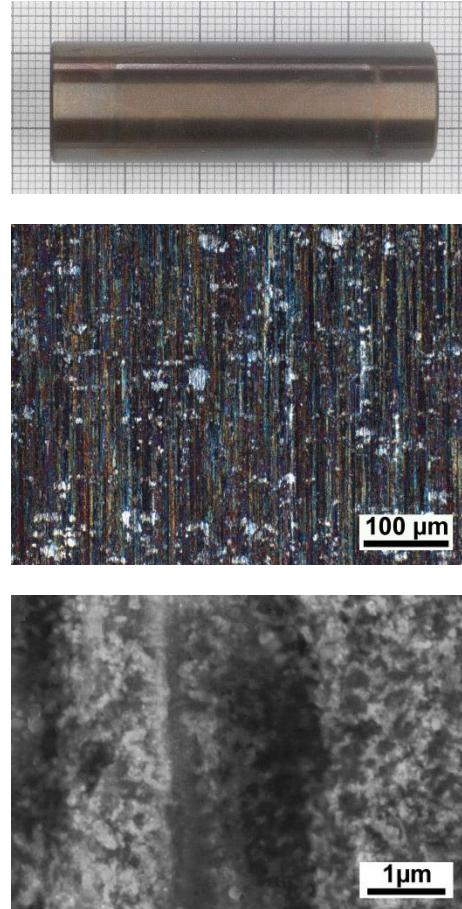


Fig. 5: Selectively oxidised $\alpha\text{-Fe}_2\text{O}_3$ specimen after 100 strokes; top: photo of tested specimen; mid position: digital light microscopic image; bottom: SEM image

After a substantial increase of the number of strokes to 5000 a thinning of the $\alpha\text{-Fe}_2\text{O}_3$ layer can be seen clearly at each magnification (Fig. 6). The photo in Fig. 6 (top) reveals that the layer has become thinner over the whole contact area. But even after 5000 strokes a remaining layer can be seen clearly also in light microscopic image (middle). In addition to the layer thinning the middle image shows scoring in the drawing direction. Also zinc pick-up from the hot dip galvanized sheet metal used was detected by EDX. The SEM image in Fig. 6 (bottom) clearly shows features that are an evidence for the existence of $\alpha\text{-Fe}_2\text{O}_3$ on the surface.

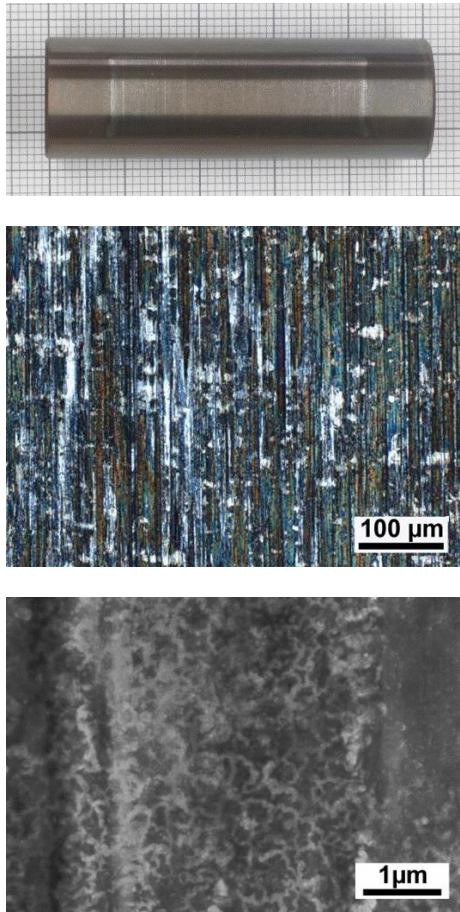


Fig. 6: Selectively oxidised $\alpha\text{-Fe}_2\text{O}_3$ specimen after 5000 strokes; top: photo of tested specimen; mid position: digital light microscopic image; bottom: SEM image

4 Summary and Outlook

Within the present study wear experiments on specimens with selectively oxidised $\alpha\text{-Fe}_2\text{O}_3$ layers were conducted with a surface pressure of 80 MPa at a process temperature of $80 \pm 5^\circ\text{C}$. The results can be summarized as follows:

It was shown that the generation of the oxide layer depends on the local composition of the surface. Surface near chromium rich precipitations were usually not covered by $\alpha\text{-Fe}_2\text{O}_3$, and thus, a complete coverage of the specimens was not realised.

Even after 5000 strokes of load (300 m of sheet metal) the friction reducing $\alpha\text{-Fe}_2\text{O}_3$ was still present on the surface, which indicates the potential of these coatings for demanding applications in dry metal forming.

Small amounts of zinc were detected on the oxidised specimen that was tested up to 5000 strokes. Whether the transfer of zinc adds to the durability of the coatings is, however, not clear yet.

It appears that oxidised surfaces might be a viable approach to realise dry metal forming as the oxides provide for low friction coefficient and feature substantial durability. Clearly, further studies need to address the tolerable maximum surface pressure between the sheet metal and specimen and how pressure affects the wear behaviour over extended periods of usage.

Acknowledgements

The authors gratefully acknowledge financial support of this study by the German Research Foundation (DFG) (grants BE1690/170-2 and MA1175/41-2) within the priority program 1676.

References

- [1] D. Wulff, D. Yilkiran, U. Holländer, D. Lützenkirchen-Hecht, R. Wagner, S. Hübner, K. Möhwald, H. J. Maier, B.-A. Behrens: Selective oxidation of 1.2379 tool steel surfaces – an approach for Dry Metal Forming, Dry Met. Forming OAJ FMT 1 (2015) 72-78.
- [2] D. Yilkiran, D. Wulff, A. Almohallami, U. Holländer, S. Hübner, K. Möhwald, B.-A. Behrens, H. J. Maier: Selectively Oxidised Tool Surfaces for Sheet Metal Forming, THe „A“ Coatings – Proc. 12th International Conference in Manufacturing Engineering (2016) 179-184.
- [3] A. Almohallami, D. Yilkiran, D. Wulff, S. Hübner, M. Vucetic, H. J. Maier, B.-A. Behrens: Numerical Modelling of the Tribology of a Selective Oxidised 1.2379 Tool Steel Surface Developed for Dry Metal Forming, Dry Met. Forming OAJ FMT 1 (2015) 91-95.
- [4] H. Kato: Severe-mild wear transition by supply of oxide particles on sliding surface. Wear 255 (2003) 426-429. DOI: 10.1016/S0043-1648(03)00077-2
- [5] H. Kato, K. Komai: Tribofilm formation and mild wear by tribosintering of nanometer-sized oxide particles on rubbing steel surfaces. Wear 262 (2007) 36-41. DOI: 10.1016/j.wear.2006.03.046
- [6] H. Kato: Effects of supply of fine oxide particles onto rubbing steel surfaces on severe-mild wear transition and oxide film formation. Tribol. Int. 41 (2008) 735-742. DOI: 10.1016/j.triboint.2008.01.001
- [7] D. Yilkiran, A. Almohallami, D. Wulff, S. Hübner, M. Vucetic, H. J. Maier, B.-A. Behrens: New Specimen Design for Wear Investigations in Dry Sheet Metal Forming, Dry Met. Forming OAJ FMT 2 (2016) 62-66.