Aluminium-alloy transfer to a CrN coating and a hot-work tool steel at room and elevated temperatures

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Abstract

During the processes of forming aluminium alloys, adhesion, and in particular the formation of the aluminium-alloy transfer on the bearing surface of the die, is one of the main reasons for the failure of tools and the poor surface quality of products. The present work was focused on the transfer initiation of an aluminium alloy (EN AW-6060) and its evolution on a coated (CrN) and an uncoated, nitrided hot-work tool steel (AISI H13) at temperatures from room temperature to 500 °C. The contact was investigated in terms of the transferred aluminium alloy surface area size on the uncoated and coated tool-steel surfaces, the topography of the wear trace and the corresponding change in the coefficient of friction.

The results show the strong dependence of the tribological properties of the hot-work tool steel and the CrN coating with respect to the aluminium alloy for the temperature together with a limited dependence on the sliding distance. At 20 °C the surface roughness was found to be the main initial cause of the aluminium alloy's transfer via mechanical interlocking. At higher temperatures (400 °C), this transfer occurred predominantly due to strong adhesion. However,

at the highest tested temperature (500 °C) the CrN coating showed a notable decrease of the adhesion with a consequent reduction in the transfer of the aluminium alloy and limited transfer-film formation.

1. INTRODUCTION

Metal-forming processes are widely used in various industries for producing larger series of different components at relatively low cost. To reduce the required forming forces and to obtain higher formability of the metals, most of the bulk-metal forming (i.e., forging, extrusion) is performed at elevated temperatures [1, 2]. At these elevated working temperatures the bearing surface of the forming die is exposed to increased mechanical and thermal stresses, high tribological loads and, in some cases, chemical attacks that lead to various types of damage, premature tool failure and poor work-piece quality [3-8].

Aluminium and its alloys have a strong tendency to adhere to the die's bearing surfaces during forming processes, which results in the substantial transfer of aluminium to the surface of the forming tool. This strong adhesion and material transfer become most problematic and evident in the processes of extrusion where materials are in the sliding contact for a longer period of time. The transferred work material becomes hardened due to the oxidation and work hardening, which then increases the forming forces and causes scratches on the subsequent work-piece surface and also, in the case of a critical volume, contributes to the interruption of forming. The transferred material must be removed and the die's bearing surface cleaned using abrasive material and chemical solutions. Furthermore, cleaning of the die surface also causes damage to the die surface and deteriorates the dimensional and geometrical tolerances of the product [9].

The tribological properties of tool steel and chromium-nitride coatings at room temperature are reasonably well known. The friction coefficient between CrN and different counter materials is up to 30 % lower in comparison to tool steel [10-13]. Furthermore, the wear resistance is up to one order of magnitude higher than the resistance of the nitrided tool steel [10-13]. On the other hand, the tribological properties of tool steels, and especially CrN coatings, at elevated temperatures have been investigated to a lesser extent. It was shown that a CrN coating is an effective method for the protection of a tool steel's surface against the abrasion of aluminium oxides (Al_2O_3), although the wear increases with a rise in the temperature [14-16]. On the other hand, it was found that in real applications and in some simulations of the hot forming of aluminium alloys, CrN coatings did not completely prevent the transfer of the aluminium alloys; despite this, the need for cleaning the tool surface was reduced by up to three times and the tool life was extended by up to four times, in comparison to a hot-work tool steel [9, 16, 17].

One of the most important mechanisms that govern the tribological contacts between the forming tools' surfaces and aluminium alloys is adhesion and the consequent transfer of the aluminium alloy to the tool surface, as was shown in several investigations [17-20]. With the occurrence of material transfer, the coefficient of friction becomes high and unsteady, as does the material mass loss [21, 22]. Some studies demonstrated that the evolution of the tribological contact's properties between the work material and the tool surface is highly dependent on the initial contact. At ambient temperatures the transfer of the aluminium alloy occurred after a few millimetres of sliding, and with a rise of the temperature the distances to material transfer were reduced [18, 19, 21, 23, 24]. With the occurrence of work-material transfer, the interface properties change dramatically and govern any further tribological behaviour. Therefore, the aim of the present work was to investigate the initial stages of the EN AW6060 aluminium

alloy's transfer onto coated and uncoated AISI H13 hot-work tool steel at elevated temperatures in the range from 20 °C to 500 °C without any lubricating media. The contact was investigated in terms of the surface area and the volume of the transferred aluminium alloy to the tool-steel and CrN surface, the topography of the wear trace and the corresponding change in the coefficient of friction.

2. EXPERIMENTAL DETAILS

2.1. Materials and surface preparation

For the purposes of this investigation nitrided hot-work tool steel (denoted as H13) and CrN-coated samples were prepared. The substrate material was an AISI H13 hot-work tool steel with the nominal composition (wt. %) 0.50 C, 0.25 Mn, 4.50 Cr, 0.55 V, 3.00 Mo and 0.20 Si. The steel samples were machined in the shape of cylinders, 10 mm in diameter and 100 mm in length, from rolled and soft-annealed bars. **Steel samples were subsequently heat treated and nitrided, which is a common procedure for hot-forming application.** Specimens were heated to the austenitizing temperature of 1025 °C, followed by gas quenching in N₂ to a temperature of 80 °C. The specimens were then pulse-plasma nitrided for 20 hours in a Metaplas Ionon HZIW 600/1000 reactor. The plasma nitriding was performed in a gas mixture of 95 vol. % H₂: 5 vol.% N₂ at 520 °C. The surface hardness (Leitz Miniload, Wild Leitz GmbH, Wetzlar, Germany) and the surface roughness (white-light interferometry, ContourGT-Ko, Bruker, Billerica, Massachusetts) were measured on all the samples. The surface hardness after nitriding was $1500 \pm 61 \text{ HV}_{0.1}$, and after the process of nitriding, the surface of the samples was reconditioned with a grinding-and-polishing process to a roughness value of $R_a = 0.036 \pm 0.006 \text{ }\mu\text{m}$.

Some of the steel samples were then coated with a CrN monolayer coating with chromium interlayer (Tecvac Ltd., Cambridge, UK) using an electron beam PVD deposition technique [25]. The coating with a hardness of 19.6 ± 0.2 GPa (measured by supplier of the coating) was applied with a thickness of approximately $3 \mu\text{m}$ and a final surface roughness $R_a = 0.038 \pm 0.004 \mu\text{m}$ was obtained.

The counter material was the commonly used aluminium alloy EN AW-6060 (denoted as 6060) with the nominal composition (wt. %) 1.00 Si, 0.70 Mn and 0.90 Mg in the form of an extruded round bar ($\phi 10 \text{ mm} \times 100 \text{ mm}$). The surface-roughness value of the aluminium-alloy samples was $R_a = 0.330 \pm 0.049 \mu\text{m}$ and the surface hardness was $72 \pm 0.3 \text{ HV}_{0.1}$.

2.2. Tribological tests

The initiation and evolution of the transfer film of the 6060 alloy on the hot-work tool steel and the CrN coating were evaluated as a function of the sliding distance and the temperature. All the tests were performed in a perpendicular cross-cylinder configuration in a single-pass dry-sliding contact with a constant load of 9 N (p_{hertz} at $20^\circ\text{C} = 0.6 \text{ GPa}$) and a speed of 0.01 m/s in air at 20°C and a relative humidity of $\sim 40\%$. The 6060 cylinder was forced to slide against the stationary CrN coating and the hot-work tool-steel cylinder, as presented in Figure 1, which ensured that the coated and uncoated steel surfaces were always in contact with a fresh and unworn aluminium-alloy specimen surface. The contact geometry configuration combined with the defined tribological parameters was selected in accordance with the contact conditions that are typical for the aluminium alloy extrusion processes.

Figure 1: Test configuration.

The tests were performed for single-pass sliding distances up to 68 mm (0 mm, 2 mm, 4 mm, 8.5 mm, 17 mm, 34 mm, 68 mm) in the temperature range from 20 °C to 500 °C. Prior to the test the aluminium-alloy samples were heated for 300 s to ensure a steady-state temperature (thermo-camera, A320, Flir, Wilsonville, Oregon) and the Type-K NiCr-Ni thermocouples (Jumo, Fulda, Germany). The 6060 aluminium-alloy samples were kept at a selected temperature for the whole duration of the test, while the hot-work tool steel and CrN specimens were kept at room temperature.

Prior to the testing all the specimens were ultrasonically cleaned in high-purity benzene, then rinsed with acetone and dried in the air. Each test was repeated at least three times to ensure a statistically relevant evaluation, and the average, together with its standard deviation, is presented in the diagrams. After each test, the surface area and the volume of the transferred aluminium alloy on the tool-steel surface, as well as the topography of the wear traces on the aluminium-alloy samples, were analysed with white-light optical interferometry (ContourGT-Ko, Bruker, Billerica, Massachusetts) and optical microscopy (Eclipse LV-150, Nikon, Tokyo, Japan). In the Figures that presents size of the surface area (Figure 3) of transferred aluminium alloy the average of the measurements with a corresponding standard deviation is presented. Selected steel and CrN-coating surfaces were investigated with a scanning electron microscope

(SEM) JEOL JSM-T330A (JEOL Ltd., Tokyo, Japan) combined with a light-element, Si, energy-dispersive X-ray detector (beryllium-window type EDS; Oxford Inst., Abingdon, UK) operated at an accelerating voltage of 10 kV, and equipped with an Inca Energy data-processing unit (Oxford Inst., Analytical Ltd., Abingdon, UK). The arrows on the SEM and the optical micrographs indicate the sliding direction of the counter surface. On selected specimens the surfaces' nanohardnesses were measured (Nano Indenter G200, Agilent technologies, California) using a Berkovich tip (TB20395 DCM) and a depth-control system of 100 nm with a Poisson's ratio $\nu = 0.35$. At least three measurements of the nanohardness were performed at different locations for each specimen to ensure relevant statistical evaluation, and the average, together with its standard deviation is presented.

3. RESULTS

3.1. Coefficient of friction

The coefficient of friction measured at six different sliding distances and temperatures of 20 °C, 400 °C and 500 °C for the contact of H13/6060 and CrN/6060 is presented in Figure 2.

Figure 2: Dependence of the coefficient of friction on the sliding distance and temperature.

The lowest coefficient of friction was measured at room temperature in the H13/6060 contact for the shortest sliding distance (2 mm), with an average value of ~ 0.52 . With an increase of the sliding distance the coefficient of friction increases and reaches its highest value (~ 1.8) after 68 mm of sliding. The value of the coefficient of friction in the contact of the CrN/6060 is slightly higher for the entire sliding distance, while the friction evolution was similar – the friction is increasing with increasing sliding distance.

When the tests were performed at higher temperatures (400 °C and 500 °C) a significant increase in the coefficient of friction was observed. Furthermore, in this temperature range the friction showed only limited dependence (slight decrease with sliding distance) on the sliding distance in the contact of the H13/6060 and almost no dependence in the CrN/6060 contact, as can be seen in Figure 2. At 400 °C the highest coefficient of friction in the entire temperature range was measured in the contact of the H13/6060, with an average value of ~ 4.5 . Moreover, in the contact of the CrN/6060 the coefficient of friction at 400 °C is ~ 33 % lower, compared to the H13/6060. On the other hand, at 500 °C similar values of the coefficient of friction were

measured for the CrN coating and the H13 steel in contact with the aluminium alloy, with an average value of ~3.5.

3.2. Surface area of the transferred aluminium alloy

Figure 3 shows the values of the size of the surface area of the aluminium alloy transferred onto the tool steel and CrN-coating surfaces for each tested temperature as a function of the sliding distance. The surface area of the transferred material showed a strong dependence on the length of the sliding distance as well as on the temperature (note the different scales in Figures 3a and 3b-c). In general, the surface area was increasing with a longer sliding distance for all the temperatures. Regardless of the temperature, the initial material transfer was observed after just 2 mm of sliding. However, as the temperature increases to 400 °C or 500 °C, an order of magnitude more aluminium material is transferred to the steel and CrN surfaces.

Figure 3: Surface area of the aluminium alloy transferred to the coated and uncoated steel surfaces at different temperatures: a) 20 °C, b) 400 °C and c) 500 °C. Note the differences in the scales.

At 20 °C the aluminium-alloy transfer occurred after the shortest sliding distance on both of the tested materials, although on the CrN it was in very small quantities (immeasurable by means of white-light interferometry), as can be seen in Figure 3a. After the initiation of the material

transfer the growth of the surface area up to 17 mm of sliding distance was similar on both the tested surfaces. However, for longer sliding distances the surface area of the transferred aluminium alloy was up to 70 % larger on the CrN coating surface.

A significant increase in the surface area (by even more than a factor of 15) of the transferred aluminium alloy was observed at temperatures of 400 °C and 500 °C, compared to 20 °C (note the differences in the scales in Figure 3). In the H13/6060 contact the material transfer occurred even for a stationary contact, without any sliding, as can be seen in Figure 3b-c. In general, a smaller area was covered with the 6060 alloy on the CrN coating, regardless of the sliding distance in comparison to the steel surface. The largest surface area of transferred material for the entire study was measured at 500 °C on the steel surface, which was valid for all the sliding distances.

3.3 Contact surfaces

3.3.1 CrN and H13 steel surfaces

At 20 °C the transfer of the aluminium alloy to the steel and the CrN surfaces was observed after just 2 mm of sliding. **The initial transfer of the alloy to either of the surfaces was observed on the surface's topographical features (i.e., in and around scratches) inside the contact area as can be seen in Figure 4.**

Figure 4: SEM micrograph of the aluminium-alloy transfer initiation to the steel surface after 2 mm of sliding distance at 20 °C. The white arrow indicates a scratch on the steel surface, while the black arrow indicates the initial aluminium-alloy transfer.

With an increasing sliding distance, the formation of the transfer film was observed as more and more of the steel and CrN contact areas were covered with the aluminium alloy (Figure 5). Although a different appearance of the transfer film is visible, a thicker and more intense transfer film was formed on the steel surface compared to the CrN surface, although it was smaller in size. However, at longer sliding distances, parts of the transfer film were entirely removed or the film was never formed fully, which can be observed in Figure 5. Namely, the EDS analysis showed the complete absence of the aluminium alloy in certain parts of the contact area. Nevertheless, the aluminium-alloy transfer films were work hardened compared to the unworn aluminium-alloy specimen surface, which was confirmed by the nano-indentation on the transfer films, indicating values of 0.75 ± 0.055 GPa vs. 1.20 ± 0.044 GPa, respectively.

Figure 5: The aluminium-alloy transfer-film formation at 20 °C after 68 mm of sliding with a marked contact area on: a) steel and b) CrN surface with the corresponding EDS spectrums.

At higher temperatures (400 °C and 500 °C) much faster and more intense aluminium-alloy transfer occurred, which is also evident from the surface area of the transfer film (Figure 3b-c).

Furthermore, aluminium-alloy transfer occurred in the stationary contact at 400 °C and 500 °C, between the aluminium alloy and the steel, as can be seen in Figure 6, which indicates enhanced adhesion between the tool steel and the aluminium alloy.

Figure 6: *The occurrence of aluminium-alloy transfer resulting from a stationary contact at 500 °C.*

The formation of the aluminium-alloy transfer film at 400 °C on the steel surface was almost instantaneous: the contact area was completely covered after just 2 mm of sliding (Figure 7a) and is then subject to only minor changes. On the other hand, the formation of the aluminium-alloy transfer film on the CrN coating at 400 °C is slower and less intense compared to the steel. Firstly, during the initial stages of sliding (2-4 mm) the aluminium alloy is adhered in and around the scratches on the CrN coating surface (Figure 7b-c), but most of the contact area is uncovered (Figure 7c), with the intact CrN surface still visible.

Figure 7: SEM observations of the aluminium-alloy transfer at 400 °C after 2 mm of sliding distance on: a) steel and b) CrN coating with c) corresponding EDS mapping micrograph of aluminium from Figure 7b.

With further sliding (8.5 mm) a complete aluminium-alloy transfer film is formed, although on regions where the transfer film is less intense the aluminium alloy build up can be observed on the topographical features, similar to the situation at 20 °C (Figure 8).

Figure 8: Aluminium-alloy transfer film on the CrN coating after 8.5 mm of sliding distance at 400 °C. Black arrows indicate the alloy build up alongside the scratches.

The formation of the transfer film on the steel and the CrN coating at 500 °C is similar, although the surface-area growth on the steel is much faster. However, on the CrN coating at longer sliding distances (34 and 68 mm) at 500 °C, parts of the transfer film were fully removed (Figure 9a). The aluminium was found in small patches stochastically on the surface, while in between them an intact CrN coating without signs of aluminium was observed (Figure 9b). The surface was confirmed to have an absence of aluminium with the EDS spectra (Figure 9c). This indicates that the adhesion between the CrN and the aluminium alloy is much weaker than between the steel and the aluminium alloy (Figure 7a).

Figure 9: a) The transfer film of the aluminium alloy on the CrN coating after 68 mm of sliding at 500 °C with b) detailed SEM micrograph of the surface where the aluminium patch was removed and c) the corresponding EDS spectrum.

3.3.2 Aluminium-alloy surface

At 20 °C, smoothening of the aluminium-alloy surface at shorter sliding distances was observed. By prolonging the sliding distance the abrasion of the aluminium-alloy surface becomes more pronounced, with the scratches oriented parallel to the sliding direction (Figure 10).

Figure 10: Aluminium-alloy specimen surface after 68 mm of sliding distance at 20 °C in contact with: a) steel and b) CrN coating. Black arrows indicate the abrasive scratches.

The topographies of the wear traces on the aluminium-alloy specimens at 400 °C for the steel and CrN coatings are very similar. The surface topography of the aluminium-alloy specimens after 2 mm of sliding distance is partially smoothed, and on the remaining part of the wear trace the early stages of a torn-like surface are observed. With increasing sliding distances up to 4 mm the torn-like surface topography occurs and the disposed patches of the aluminium alloy become apparent (Figure 11). By prolonging the sliding distance the torn-like surface becomes more pronounced for both the steel and the CrN.

Figure 11: *Aluminium alloy specimen surface after 2 mm of sliding distance at 400 °C in contact with the CrN coating with the marked disposed patches.*

At 500 °C in the case of the H13/6060 contact a very similar wear-trace topography as at 400 °C was found with the torn-like surface and the smeared disposed patches of aluminium alloy (Figure 12a). On the other hand, a completely different wear behaviour and consequent topography of the wear trace on the aluminium-alloy samples was found in the contact of the CrN/6060. The surface of the wear trace is completely smoothened and is subject to no change up to 34 mm of sliding distance (Figure 12b). With further sliding, the smoothening is accompanied by a torn-like surface.

Figure 12: Aluminium-alloy specimen surface after 34 mm of sliding distance at 500 °C in contact with a) steel and b) CrN coating.

4. DISCUSSION

The contact of the nitrided AISI H13 hot-work tool steel and the CrN coating against the EN AW-6060 aluminium alloy showed a highly temperature-dependent tribological behaviour, as is schematically presented in Figure 13.

Figure 13: Characteristic evolution of the a) coefficient of friction and b) the surface area of the transferred aluminium alloy as a function of temperature.

At 20 °C the coefficient of friction has lower values (0.5–2) and the friction is increasing with a prolonging of the sliding distance for both tested materials (Figure 2). In general, an approximately 20 % lower coefficient of friction was measured in the contact of the H13/6060

compared to the CrN/6060 contact. The value and evolution of the friction (Figure 13a) correspond well with the evolution and the size of the transferred aluminium alloy (Figure 13b), which is increasing with the sliding distance (Figure 3a). The initial cause of the aluminium-alloy transfer at 20 °C was found to be surface irregularities, such as scratches and defects (Figure 4), which by prolonging the sliding distance results in the formation of a transfer film. However, the transfer film (Figure 5) can be in parts fully removed during sliding because of the lack of adhesion, indicating a mechanical interlocking as the predominant transfer mechanism. With the transfer-film formation the transferred aluminium alloy also gets work-hardened, by as much as 40 %, causing scratches on the wear trace of the aluminium-alloy specimens (Figure 10).

The coefficient of friction at elevated temperatures (400–500 °C) initially reaches very high values and is decreasing with a prolongation of the sliding distance in the H13/6060 contact, while in the CrN/6060 contact a relatively constant friction through the whole 68 mm of sliding is observed (Figures 2, 13a). At 400 °C a 30 % lower coefficient of friction was measured in the contact of the CrN/6060 compared to the H13/6060. This behaviour correlates with the size (Figure 3b) and especially with the rapid initial growth of the surface area of the transferred aluminium alloy (Figures 3b, 7), where faster formation and a larger surface area of the transfer film were found on the H13 steel surface compared to the CrN coating (Figures 7b-c, 8). Even more, the aluminium-alloy transfer occurred on the steel surface even in a stationary contact, i.e., without any sliding (Figure 6), indicating adhesion as being the main aluminium-alloy transfer mechanism on the steel [24]. This strong adhesion between the aluminium alloy and the steel could also explain the higher initial values of the coefficient of friction compared to the CrN coating.

Similar behaviour was observed at 500 °C in the H13/6060 contact, while on the CrN surface the transfer-film formation was incomplete (Figure 9), indicating lower adhesion of the aluminium alloy on the surface of the CrN coating. The slower aluminium-alloy transfer-film growth at 400 °C and the incomplete transfer-film coverage at 500 °C could be a consequence of the accelerated formation of chromium oxides (Cr_xO_y) at higher temperatures, which form a passivating layer that reduces the adhesion between the alloy and the coating, as was reported by some authors [14, 15, 26]. However, despite the reduced adhesion between the CrN coating and the 6060 alloy, the coefficient of friction at 500 °C is similar for both tested materials (Figure 2). This is in agreement with the reduced yield strength of the aluminium alloy at 500 °C (by a factor of 30 compared to the yield strength at 20 °C [24]) and, consequently, a reduced shear resistance of the aluminium alloy. Therefore, the friction force at these high temperatures derives from the bulk plastic deformation of the aluminium-alloy sample [24], rather than the adhesion component of the friction. Nevertheless, the reduced adhesion of the aluminium alloy on the CrN coating at 500 °C is favourably reflected in the wear-trace topography, which became very smooth (Figure 12b), and is especially important with respect to the hot-forming work-piece surface quality in a real application.

5. CONCLUSIONS

We have investigated the effect of sliding distance and temperature in the range from 20 to 500 °C on the initiation and evolution of an aluminium-alloy EN AW-6060 on a nitrided AISI H13 hot-work tool steel and a CrN coating.

1. The principal initial cause for the transfer of the aluminium alloy at 20 °C was found to be the topography irregularities, both on the hot-work tool steel and the CrN surface. In

contrast at higher temperatures (400 and 500 °C) the transfer of the aluminium alloy occurred predominantly due to strong adhesion.

2. At 20 °C, a 20 % higher coefficient of friction was obtained in the 6060/CrN contact compared to the H13/6060 contact. In contrast, at 400 °C, a higher friction was obtained in the H13/6060 than in the 6060/CrN contact, while at 500 °C very similar values for both the H13/6060 and 6060/CrN contacts were measured.
3. The evolution of the friction, which is increasing with sliding distance at 20 °C, was very similar for both the tested materials and was increasing with the sliding distance. The friction behaviour, however, changed with the increase of the temperature to 400 °C and 500 °C. Namely, in the 6060/CrN contact the friction was constant and the sliding distance was independent. **On the other hand, in the H13/6060 contact the coefficient of friction at shorter sliding distances even decreased with the increasing sliding distance.**
4. The size of the surface area of the transferred aluminium alloy correlates with the coefficient of friction for the tested materials at 20 °C. A smaller surface area and a lower friction were measured on the H13 steel surface, while conversely, at higher temperatures (400–500 °C), a smaller surface area and friction were measured on the CrN coating.
5. At higher tested temperatures (400–500 °C) the extensive plastic deformation of the aluminium-alloy is the main contribution to the high values of the coefficient of friction, rather than the interface properties between the aluminium alloy and the steel or CrN, which is the case for low-temperature sliding.
6. The aluminium-alloy transfer mechanism is reflected in the topography of the wear trace of the aluminium alloy. At 20 °C, there is a mainly smooth surface with some scratches that are induced by the work-hardened aluminium-alloy transfer film. In contrast, at higher temperatures, where a strong adhesion is present, the torn-like surface on the aluminium

alloy dominates. If the adhesion-based transfer film is minimised (CrN at 500 °C) the desired, smooth, aluminium-alloy surface can be formed.

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