



# The effect of temperature and sliding distance on coated (CrN, TiAlN) and uncoated nitrided hot-work tool steels against an aluminium alloy



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## ABSTRACT

Adhesion and, in particular, the transfer of aluminium alloys to the bearing surface of a die are two of the main reasons for tool failure and the poor surface quality of products, especially at elevated temperatures. The present work was focused on the EN-AW6060 aluminium alloy's transfer initiation and the evolution to an AISI H13 hot-work tool steel, as well as CrN and TiAlN coatings in a cross-cylinder, single-pass, dry-sliding contact at room (20 °C) and elevated temperatures (300–500 °C). The contact was investigated in terms of the surface area and volume of the transferred aluminium alloy to the tool steel's surface, the topography of the wear trace and the corresponding change in the coefficient of friction. The results show a strong dependence of the tribological properties of the investigated materials on the temperature and only a limited dependence on the sliding distance, especially for the TiAlN coating. At room temperature the lowest coefficient of friction and the smallest amount of material transfer were measured for the TiAlN coating. At higher temperatures both the CrN and TiAlN coatings showed similar friction values and amounts of transferred aluminium alloy, while the nitrided hot-work tool steel exhibited an inferior tribological performance.

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## 1. Introduction

Aluminium alloys have a high tendency to transfer to a die's bearing surfaces, especially during warm- and hot-forming processes [1–3]. This strong adhesion and the transfer of the aluminium alloy become most problematic and evident in the processes of extrusion, where the materials are in a sliding contact for a longer period of time [2]. The transferred aluminium alloy increases the forming force, and it becomes hardened due to oxidation and work hardening, thereby causing scratches on the subsequent workpiece surface [4]. Due to the transferred aluminium alloy the forming process must be interrupted and the die's bearing surface, cleaned using silicon carbide abrasive paper and, on occasions, chemical solutions must be used, which can cause damage to the die surface and deteriorate the dimensional and geometrical tolerances of the product [5]. To improve the service life of the forming tools and the quality of the product, the surfaces of the tools can be coated with thin, hard coatings, which can operate at high temperatures [6–20]. With the use of protective layers such as CrN and TiAlN in the contact, and with different counter materials, the coefficient of friction can be reduced by as much as 30%, compared to the hot-work tool steel that is typically used in forming applications [5,21–32]. Furthermore, the wear resistance is up to an order of magnitude higher than the resistance of the nitrided tool

steel [5,21–32]. However, in our previous studies [33–36] we demonstrated that the evolution of the tribological properties of the interface between the aluminium alloy and the “tool” surface is very dependent on the initial contact. At ambient temperatures the transfer of the aluminium alloy occurred after a few millimetres of sliding; however, with a rise in the temperature, the distances necessary for aluminium-alloy transfer were even shorter. With the occurrence of work-material transfer the interface properties change dramatically and tend to govern any subsequent tribological behaviour. Therefore, the aim of the present work was to investigate and compare the ability of the AISI H13 hot-work tool steel, as well as the CrN and TiAlN coatings, to prevent the initiation and evolution of the transfer of the aluminium alloy. The contact was investigated in terms of the surface area and the volume of the transferred aluminium alloy to the tool steel's 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 purpose of this investigation, nitrided hot-work tool steel (denoted as H13), CrN- and TiAlN-coated samples were prepared. The steel samples were machined from rolled and soft-annealed round bars of AISI H13 hot-work tool steel (nominal composition wt%: 0.50 C, 0.25 Mn, 4.50 Cr, 0.55 V, 3.00 Mn and

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0.20 Si) in the shape of cylinders, 10 mm in diameter and 100 mm in length. Steel samples were subsequently heat treated and nitrided, which is a common procedure for hot-forming application. 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 ( $\phi 10$  mm  $\times$  100 mm).

Hardness and roughness of steel and aluminium samples were evaluated after different preparation steps. Vickers hardness was measured using the hardness tester (Leitz Miniload, Wild Leitz GmbH, Wetzlar, Germany) at the load of 981 mN ( $HV_{0.1}$ ). At least 10 measurements were performed on each material and the average with a corresponding standard deviation is presented. The surface roughness was measured on steel, aluminium and coated samples. 3D optical microscope using white light (ContourGT-Ko, Bruker, Billerica, Massachusetts). All measurements were done in accordance to the standard EN ISO 4288. For all materials at least 10 measurements were performed and the average with a corresponding standard deviation is presented.

After initial machining the steel specimens were heated to an austenitizing temperature of 1025 °C, followed by gas quenching in  $N_2$  to a temperature of 80 °C (Table 1), which resulted in a hardness of  $553 \pm 6 HV_{0.1}$ . The specimens were then pulse-plasma nitrided [33] for 20 h in a Metaplas Ionon HZIW 600/1000 reactor. The plasma nitriding was performed in a gas mixture of 95 vol%  $H_2$ : 5 vol%  $N_2$  at 520 °C, which resulted in a surface hardness of  $1500 \pm 61 HV_{0.1}$ . The resulting diffusion zone was approximately 100  $\mu$ m deep. After the process of nitriding, the surface of the samples was reconditioned with a grinding-and-polishing process to a final roughness value of  $R_a = 0.036 \pm 0.006 \mu$ m.

One-third 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 [36]. The coating with a hardness of  $19.6 \pm 0.2$  GPa (measured by supplier of the coating) was applied with a thickness of approximately 3  $\mu$ m and a final surface roughness  $R_a = 0.038 \pm 0.004 \mu$ m was obtained.

The last third of the steel samples were coated with a commercially available TiAlN monolayer coating (Tecvac Ltd., Cambridge, UK) with titanium interlayer. The coating with a thickness of approximately 3  $\mu$ m and a hardness of  $30.1 \pm 0.3$  GPa (measured by supplier of the coating) was applied with a magnetron sputtering PVD technique [37]. The final surface roughness of the TiAlN-coated samples was  $R_a = 0.040 \pm 0.005 \mu$ m, as presented in Table 2.

The surface-roughness value of the aluminium-alloy counter parts was  $R_a = 0.330 \pm 0.049 \mu$ m and the surface hardness was  $72 \pm 0.3 HV_{0.1}$ .

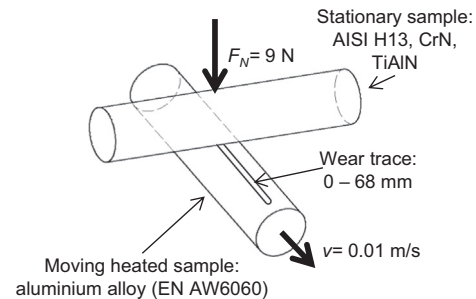
## 2.2. Tribological tests

The ability of the investigated hot-work tool steel, the CrN and the TiAlN coatings to prevent the initiation and evolution of the transfer of the aluminium was 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 °C  $\approx$

**Table 2**

Hardness and surface-roughness parameters of the samples.

Sample treatment	H13 Nitrided hot-work tool steel	CrN CrN monolayer coating	TiAlN TiAlN monolayer coating
Hardness	$1500 \pm 61 HV_{0.1}$	$19.6 \pm 0.2$ GPa	$30.1 \pm 0.3$ GPa
$R_a$ , $\mu$ m	$0.036 \pm 0.006$	$0.038 \pm 0.004$	$0.040 \pm 0.005$
$R_{qz}$ , $\mu$ m	$0.049 \pm 0.009$	$0.049 \pm 0.005$	$0.052 \pm 0.007$
$R_z$ , $\mu$ m	$0.810 \pm 0.109$	$0.804 \pm 0.082$	$1.354 \pm 0.212$
$R_{ku}$	$3.752 \pm 0.190$	$3.748 \pm 1.165$	$4.153 \pm 1.899$
$R_{sk}$	$-1.025 \pm 0.094$	$-0.695 \pm 0.243$	$-0.660 \pm 0.924$



**Fig. 1.** Test configuration.

0.6 GPa) and a speed of 0.01 m/s. A 6060 cylinder was forced to slide against the stationary H13, CrN and TiAlN cylinders, as presented in Fig. 1, which ensured that the coated and uncoated steel surfaces were always in contact with a fresh and unworn aluminium-alloy specimen surface.

The tests were performed for sliding distances of 0 mm, 2 mm, 4 mm, 8.5 mm, 17 mm, 34 mm and 68 mm, where after reaching the required distance, a completely new test with a longer distance has started. The temperatures varied at 20 °C, 300 °C, 400 °C and 500 °C. Prior to the test, the 6060 samples were heated in the sample holder (holder with calibrated ceramic heaters operated by a thermostat and thermocouples on the surface of aluminium alloy sample) for 300 s to the selected temperature to ensure a uniform temperature distribution for the whole sample, which was monitored with a thermo-camera (A320, Flir, Wilsonville, Oregon) and thermocouples. The 6060 aluminium-alloy samples were kept at a selected temperature for the whole duration of the test, while the H13, CrN and TiAlN specimens were kept at room temperature.

The tests were performed in air at 20 °C and a relative humidity of  $\sim 40\%$ . During the testing the coefficient of friction was monitored as a function of the sliding distance. Prior to the testing all the specimens were ultrasonically cleaned in high-purity benzene, then rinsed with acetone and dried in 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 (Fig. 3) and volume (Fig. 5) of transferred aluminium alloy the average of the measurements with a corresponding standard deviation is presented. The average transfer film thickness was calculated from the measured volume and surface area of the aluminium alloy transferred to the tested materials after 68 mm of sliding distance. The average of the calculated thicknesses with a corresponding standard deviation is

**Table 1**

Parameters of the heat treatment of the AISI H13 hot-work tool steel [33].

Treatment	Temperature, °C	Time, h
Preheat	850	/
Austenitizing	1025	0.5
Gas quenching in $N_2$	80	/
Tempering 1	540	3.5
Tempering 2	600	3.5

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