







indentations distinguish between different adhesion classes (HF) of HF 1 (very good adhesion) and HF 6 (insufficient adhesion).

### 3 Results and discussion

Exemplary cross section fracture of the HPPMS (Cr,Al)N coating (sample 10) is shown in Fig. 6. Cross section fractures of the other samples confirmed that the morphology does not change according to pre-treatment if the parameters of the coating deposition kept fixed. It can be clearly seen that the coating has a fine columnar morphology.

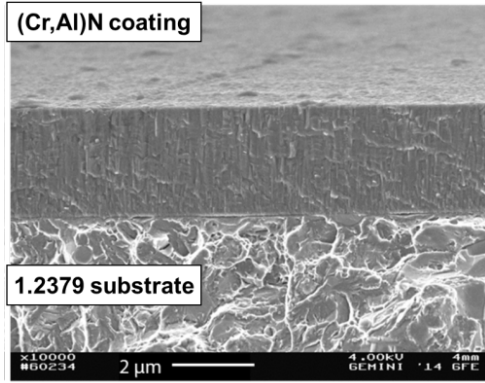


Figure 6: SEM cross section fracture of the (Cr,Al)N coating (S10).

The coating thickness is 2.60  $\mu\text{m}$ , which corresponds to a deposition rate of 0.52  $\mu\text{m}/\text{h}$ . The chemical composition of the deposited coatings as measured by GDOES shows no differences regarding to pre-treatment as well. Exemplary results are as follows:  $X_{\text{Cr}} = 31$  at.-%,  $X_{\text{Al}} = 11$  at.-% and  $X_{\text{N}} = 58$  at.-% (sample 10), see Fig. 7. Cr:Al ratio is 74:26 (at.-%).

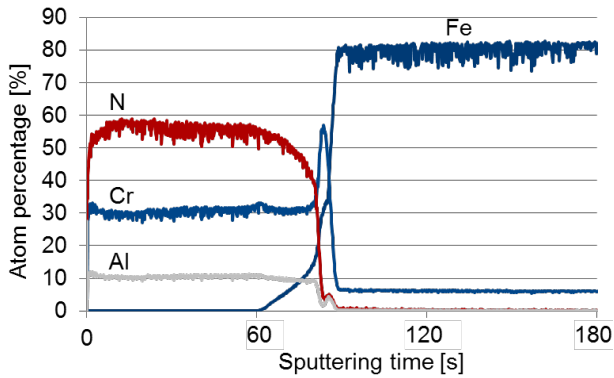


Figure 7: GDOES investigation of the (Cr,Al)N coating (S10).

Regarding the mechanical properties as universal hardness (HU) and the modulus of indentation ( $E_{\text{IT}}$ ), it must be stated that the  $\text{HU}^3/E_{\text{IT}}^2$  ratio characterizes the resistance of the material to plastic deformation [18]. Therefore, a high HU and a low  $E_{\text{IT}}$  are aimed for a tribological application. In the case of (Cr,Al)N matrix the mechanical properties are strictly dependent on the Cr:Al ratio. The determination is done on the basis of previous investigations [19]. Mechanical properties do not show differences regarding the pre-treatment art

either. Exemplary results (sample 10) are as follows:  $\text{HU} = 28.6 \pm 2.8$  GPa and  $E_{\text{IT}} = 365.1 \pm 25.8$  GPa.

Scratch tests on all samples were done in order to evaluate the adhesion strength between the substrates and the specimens. Critical loads at which the coating failure occurs were determined and compared with each other. The specimens which show better adhesion strength between the coating and the substrate in each variation group are shown in Fig. 3 and 4 as bold and italic characters. Figure 8 shows exemplary an investigation of scratch tracks and a Rockwell indentation test by means of light microscopy. The arrow indicates the direction of the scratches. Table 1 summarizes the results of all specimens regarding adhesion strength. As mentioned before, the last at which the plastic deformation occurs at the scratch track's edge is determined by  $L_{\text{C1}}$ . This value is not interesting for the purposed application. Therefore, the focus was on the  $L_{\text{C2}}$  and  $L_{\text{C3}}$  loads.

		Variation	Sample	$L_{\text{C2}}$ [N]	$L_{\text{C3}}$ [N]	Rockwell HF	
mf Ar ion etching	Duration	20 min	S1	30	30	2	
		40 min	S2	40	40	2	
		60 min	S3	40	50	1	
	Voltage	550 V	S4	30	30	2	
		600 V	S5	30	40	2	
		650 V	S6	40	50	1	
	Ar-Pressure	250 mPa	S7	40	50	2	
		300 mPa	S8	40	40	2	
		350 mPa	S9	30	40	2	
HPPMS Cr ion etching	dc bias	500 Hz	S10	20	30	3	
		350 Hz	S11	30	30	2	
		200 Hz	S12	40	40	2	
	Power	2,5 kW	S13	20	30	3	
		3,5 kW	S14	30	30	2	
		4,5 kW	S15	40	40	2	
	HPPMS bias	Pulse length	50 $\mu\text{s}$	S16	30	40	3
		100 $\mu\text{s}$	S17	40	40	2	
		200 $\mu\text{s}$	S18	40	50	2	
Offset	with	S19	50	60	3		
	without	S20	40	50	2		

Table 1: Investigation of the specimens regarding adhesion strength between (Cr,Al)N coating and substrate.

The in Fig. 8 observed critical scratch loads  $L_{\text{C2}}$  and  $L_{\text{C3}}$  for this compound are 40 N and 50 N, respectively. At  $L_{\text{C2}}$  interfacial decohesion of the coating along the

scratch track borders occurs. At 50 N an adhesive failure of the coating is visible.

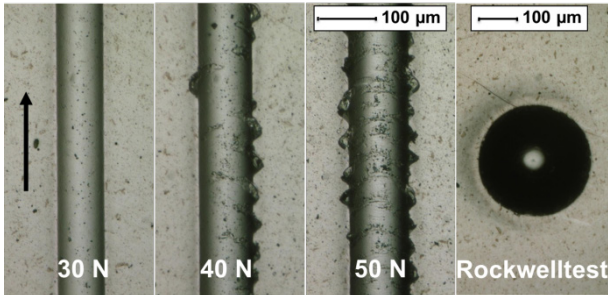


Figure 8: Scratch tracks at different loads and rockwelltest of the (Cr,Al)N coating (S6).

Considering the results in Table 1, the (Cr,Al)N coating exhibits the ideal critical loads on the X155CrMoV12 substrate with the variation of the following parameters in the mf argon ion etching: a duration of 60 min, a voltage of 650 V and a Ar pressure of 250 mPa. This pre-treatment will be used for the future studies on the further development of the (Cr,Al)N coating.

All Rockwell indentations exhibit adhesion classes between HF 1 and HF 3 regarding the classification by VDI guideline 3198 and these are all considered as acceptable. It can be said, that the HF classes of the coatings with Cr ion etching pre-treatment are lower comparing to the samples pre-treated with mf gas etching. But one of the aspects of the Cr ion etching treatment by HPPMS technology is the possibility of metal ion implantation into the surface which can lead to improvement of bonding force between the coating and the substrate surface [20]. In order to investigate this effect, an uncoated X155CrMoV12 specimen was sheltered in one half to prevent any treatment and the other half was Cr etched with the same parameters as by sample 19 (HPPMS Cr ion etching with HPPMS bias and offset configuration). Afterwards, the transition zone on the surface was analyzed by EDX line-scan, see Fig. 9. Existence of Cr on the etched surface is proven. Also SEM cross section fractions confirmed this observation, showing a thin Cr top-layer with a thickness of about 200 nm, see Fig. 10.

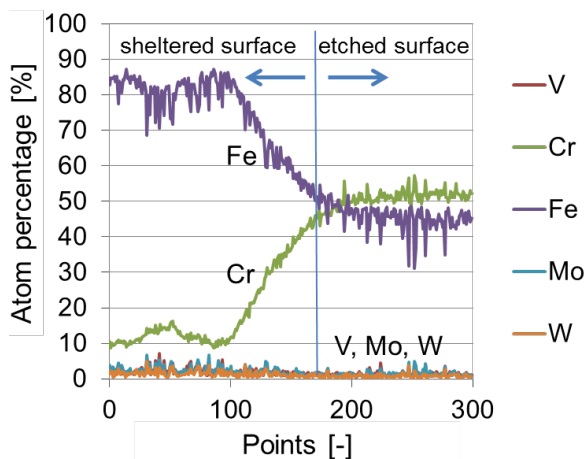


Figure 9: EDX line-scan of the sheltered and etched surface.

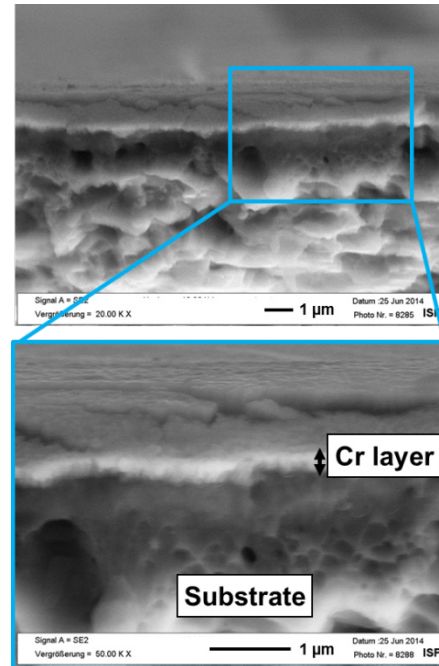


Figure 10: SEM cross section fracture of the etched surface.

#### 4 Conclusion and outlook

The absence of lubricants in dry metal forming significantly contributes to waste reduction in manufacturing processes and to the goal of a lubricant-free factory. However, the avoidance of lubricant usage goes along with the requirement that the dry tribological system or coating has to withstand the increased tribological loads. In this contribution a plasma etching process is developed for the (Cr,Al)N PVD tool coating to ensure a sufficient adhesion strength on the tool material.

Since the results in this study only give a first impression of a very recent research, there remain topics to finish in the future. Firstly, (Cr,Al)N development by means of HPPMS technology will be completed. After that, the self-lubricating disulfides will be embedded into the (Cr,Al)N matrices. Application oriented wear tests using the Pin-On-Disc (IOT) and a novel Pin-On-Cylinder tribometer (WZL) will be performed to analyze the friction behavior which will constitute an important extension of this work. Also, the advances in surface structures on workpieces will be investigated to provide friction reducing surfaces in dry metal forming by experimental studies of surface structures by WZL in order to identify friction and, thus, tool load reducing surfaces compared with non-structured workpieces. Synthesized, these two approaches from two institutions will achieve a lubricant free cold forging.

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## References

- [1] F. Klocke: Manufacturing processes 4 - Forming, Springer, (2013).
- [2] H. Czichos, K. Gerschwiler: Tribologie-Handbuch. Reibung und Verschleiß. Wiesbaden: Vieweg, (2007).
- [3] E. Lugscheider, K. Bobzin, C. Pinero, F. Klocke, T. Massmann: Development of a superlattice (Ti,Hf,Cr)N coating for cold metal forming applications. Surface and Coatings Technology, 177-178 (2004) 616-622.
- [4] K. Bobzin, N. Bagcivan, P. Immich, C. Warnke, F. Klocke, C. Zeppenfeld, P. Mattfeld: Advancement of a nanolaminated TiHfN/CrN PVD tool coating by a nano-structured CrN top layer in interaction with a biodegradable lubricant for green metal forming. Surface and Coatings Technology, 203 20-21 (2009) 3184-3188.
- [5] F. Vollertsen, F. Schmidt: Dry Metal Forming: Definition, Chances and Challenges. Int. J. Precision Engineering and Manufacturing – Green Technology 1/1 (2014) 59–62.
- [6] N. Bay, A. Azushima: Environmentally benign tribo-systems for metal forming. CIRP Ann. Manuf. Technol. 59/2 (2010) 760–780.
- [7] E. Lugscheider, K. Bobzin, M. Beckers, M. Burckhardt, Untersuchung der Versagensmechanismen von PVD-Schichtsystemen in umweltverträglichen Kaltumformungs-Tribosystemen, Report and Conference Proceedings of the GfT-Conference, Göttingen, Germany, 2001, pp. 21/1–9. Available from <<http://www.gft-ev.de>>
- [8] A. Bruzzone, H. Costa, P. Lonardo, D. Lucca: Advances in engineered surfaces for functional performance. CIRP Ann. Manuf. Technol. 57 (2008) 750–769.
- [9] J. Lin, B. Mishra, J.J. Moore, W.D. Sproul: Microstructure, mechanical and tribological properties of  $Cr_{1-x}Al_xN$  films deposited by pulsed-closed field unbalanced magnetron sputtering (P-CFUBMS). Surface and Coatings Technology, 201 (2006) 4329.
- [10] K. Bobzin, E. Lugscheider, M. Maes, P.W. Gold, J. Loos, M. Kuhn: High-performance chromium aluminium nitride PVD coatings on roller bearings. Surface and Coatings Technology, 188–189 (2004) 649-654.
- [11] K. Bobzin, E. Lugscheider, R. Nickel, N. Bagcivan, A. Krämer: Wear behavior of  $Cr_{1-x}Al_xN$  PVD-coatings in dry running conditions. Wear, 263 (2007) 1274-1280.
- [12] K. Bobzin, N. Bagcivan, M. Ewering, R.H. Brugnara, S. Theiß: DC-MSIP/HPPMS (Cr,Al,V)N and (Cr,Al,W)N thin films for high-temperature friction reduction. Surface and Coatings Technology, 205 8–9 (2011) 2887-2892.
- [13] J. Alami, S. Bolz, K. Sarakinos: High power pulsed magnetron sputtering: Fundamentals and applications. Journal of Alloys and Compounds, 483 1–2 (2009) 530-534.
- [14] K. Macak, V. Kouznetsov, J. Schneider, U. Helmersson, I. Petrov: Ionized sputter deposition using an extremely high plasma density pulsed magnetron discharge, Journal Of Vacuum Science & Technology A-Vacuum Surfaces And Films, 18 (2000) 1533-1537.
- [15] W.C. Oliver, G.M. Pharr: An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. J. Mater. Res., 7 6 (1992) 1564-1583.
- [16] DIN EN 1071-3:2005 Advanced technical ceramics Methods of test for ceramic coatings Part 3: Determination of adhesion and other mechanical failure modes by a scratch test.
- [17] Verein Deutscher Ingenieure Normen, VDI 3198, VDI-Verlag, Dusseldorf (1991).
- [18] J. Musil: Hard and superhard nanocomposite coatings. Surface and Coatings Technology, 125 (2000) 323-330.
- [19] N. Bagcivan, K. Bobzin, S. Theiß: Comparison of  $(Cr_{0.75}Al_{0.25})N$  Coatings Deposited by Conventional and High Power Pulsed Magnetron Sputtering. Contrib. Plasma Phys., 52 (2012) 601–606.
- [20] A.P. Ehiasarian, W.D. Munz, L. Hultman, U. Helmersson, I. Petrovic: High power pulsed magnetron sputtered CrN<sub>x</sub> films. Surface and Coatings Technology, 163-164 (2003) 267.