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Accelerated Tribo-Films Formation in Complex Adaptive Surface-Engineered Systems under the Extreme Tribological Conditions of Ultra-High-Performance Machining

German S. Fox-Rabinovich [1], Iosif S. Gershman [2] and Jose Luis Endrino [3,*]

[1] Department of Mechanical Engineering, McMaster University, Hamilton, ON L8S 1J7, Canada
[2] Joint Stock Company "Railway Research Institute", 129851 Moscow, Russia
[3] Department of Engineering, Universidad Loyola Andalucia, Av de las Universidades s/n, 41704 Sevilla, Spain
* Correspondence: jlendrino@uloyola.es

Abstract: This study investigates accelerated physical–chemical processes in a complex adaptive surface-engineered system represented by a nano-multilayer TiAlCrSiYN/TiAlCrN PVD coating under the extreme tribological conditions of ultra-high-performance dry machining of hardened H 13 tool steel. These processes are similar to the different catalyzing phenomena. Experimental results of tool life vs. wear rate, SEM/TEM data of the worn surfaces, XPS and EDS data of tribo-films formed on the friction surfaces, and chip surface morphology are presented in this study. The corresponding relationships between self-organization, self-organized criticality, and various catalyzing phenomena were evaluated on the basis of the accrued data. A method of enhancing these processes through the variation of machining conditions is also outlined, which resulted in the improvement of coated tool life by 35%.

Keywords: self-catalyzing and auto-catalyzing reactions; nano-multilayer PVD coating; self-organization; self-organized criticality; extreme tribological conditions

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Citation: Fox-Rabinovich, G.S.; Gershman, I.S.; Endrino, J.L. Accelerated Tribo-Films Formation in Complex Adaptive Surface-Engineered Systems under the Extreme Tribological Conditions of Ultra-High-Performance Machining. *Lubricants* **2023**, *11*, 221. https://doi.org/10.3390/lubricants11050221

Received: 30 March 2023
Revised: 3 May 2023
Accepted: 9 May 2023
Published: 15 May 2023

1. Introduction

In recent times, various catalyzing reactions have caught the attention of researchers. It was discovered that some reactions are self-catalyzing [1], wherein a sequence of events creates by-products that accelerate said reaction. The greater the variety of chemicals and the higher the temperature in the system, the greater the likelihood that a self-catalyzing reaction will occur [1–4]. The other discovered catalyzing chemical reaction is an autocatalytic reaction in which at least one of the reactants is also a product [4–6]. It is known from the literature [7] that self-organization under far-from-equilibrium conditions leads to the growth of the system's complexity. Such structures could emerge during collective (synergistic) interactions. It would be of significant interest to investigate these processes under strongly non-equilibrium conditions, especially for systems operating on the edge of chaos [8].

The edge of chaos is a dynamic state at the boundary between order and chaos [8]. The most straightforward method of investigating the outlined complex processes under extreme conditions is through specific case studies. One such case study is presented in this paper. Coming from the field of tribology, a typical extra high-speed machining process develops on the edge of chaos [9,10]. The analyzed complex adaptive system is represented by specially designed thin-film nanomaterials (Physical vapor deposited (PVD) coatings), capable of withstanding such extreme conditions [11]. Multiple closely interrelated processes, in particular that of self-organization, and self-organized criticality were investigated to gain a better understanding of the observed complex phenomena [12].

Before further discussion proceeds, a few points need to be clarified. First of all, the temperature in the friction zone is extremely high during extra-high-performance ball-nose end milling of hardened tool steels, ranging from 1000 °C [13] and above, and is accompanied by heavy loads between 1.5 and 2 GPa [8]. Secondly, the severity of frictional conditions depends on the extent of seizure between the tool and the workpiece, which is typical for the cutting process [9]. Seizure is a catastrophic tribological process [14] in which the asperities between contacting frictional bodies (tool and workpiece) become interlocked and cease relative motion. Therefore, the ensuing friction is characterized by the shearing of the formed strong adhesive bonds [15]. Under the studied cutting conditions, deformation can become extremely severe. The cutting tool and workpiece move in relation to each other under varied seizure conditions across the entire rake surface. This is possible due to the strong plastic deformation of the workpiece material that results in its shearing [9]. Severe plastic deformation also generates intensive heat within the thin layers closest to the tool/chip interface [9]. Modern cutting tool materials, specifically hard thin-film coatings, are generally strong enough to withstand these extreme tribological conditions. Tribo-film formation during cutting involves several phases. First, the aforementioned friction-generated heat initiates the mass transfer of corresponding elements within the coating layer to the friction surface. This process is strongly accelerated by the adaptive response of the analyzed thin-film PVD coating layer to an initial, strongly non-equilibrium state [10]. The next stage in this complex process is non-spontaneous tribo-oxidation caused by the interaction of the coating layer with the surrounding oxygen [7], which leads to the formation of extremely thin (nano-scale) layers of tribo-films on the friction surface [15–23]. These processes are directly related to self-organization with the formation of dissipative structures [24,25]. Intensive processes with negative entropy production are undergoing in this way. Under the examined conditions, the processes that develop on the friction surface are more complicated, since the workpiece and the tool are in seizure. Moreover, self-organized critical and self-organization processes are occurring in sequence [10].

To increase the wear resistance of the analyzed complex adaptive surface-engineered system, the chief aim of the present research study is to investigate catalyzing processes from the viewpoint of self-organization phenomena.

2. Experimental Section

A state-of-the-art TiAlCrSiYN/TiAlCrN multilayer physical vapor deposited (PVD) coating was studied.

The nano-multilayered $Ti_{0.2}Al_{0.55}Cr_{0.2}Si_{0.03}Y_{0.02}N/Ti_{0.25}Al_{0.65}Cr_{0.1}N$ coating was deposited using $Ti_{0.2}Al_{0.55}Cr_{0.2}Si_{0.03}Y_{0.02}$ and $Ti_{0.25}Al_{0.65}Cr_{0.1}$ targets fabricated by a powder metallurgical process on a WC–Co cemented carbide ball-nose end-mill substrates in an R&D-type hybrid PVD coater (Kobe Steel Ltd., Hyogo, Japan) using a plasma-enhanced arc source. The deposition parameters are shown in Table 1.

Table 1. Deposition parameters of the studied coatings.

Heating Procedure	Reactant Gas	Gas Pressure, during Deposition, Pa	Target Size, mm	Current of Arc Sources, A	Bias Voltage, V	Substrate Rotation during Deposition, rpm
Samples were heated up to about 500 °C and cleaned through an Ar ion etching process	Pure N_2 gas was fed to the chamber	4	100 mm diameter × 16 mm thick target Two targets were used for coating deposition.	150	100	5

The characteristics of the coating are shown in Table 2.

An XRD X-ray Diffraction System from Proto Manufacturing Limited (LaSalle, ON, Canada) with Cu Kα (1.544 Å) radiation was used to determine the crystal structure. XRD studies of the analyzed coatings were also carried out to identify the formed phase values, using a Siemens D500 diffractometer (Garching, Germany) with a Cu Kα tube in the θ/2θ mode. The grain size (g.s.) was determined for the main (200) orientation. The standard reference value of the (200) peak in the TiN FCC B1 type structure was 42.633, as specified in the Joint Committee on Powder Diffraction (JCPDS, 87-0633). A scanning electron microscope (SEM) (Vega 3-TESCAN, Libušina tˇr., Brno–Kohoutovice, Czech Republic) was used for the detailed inspection of the surface morphology of the studied cutting tools and chips.

Table 2. The structural and micro-mechanical characteristics of TiAlCrSiYN/TiAlCrN multilayer coatings [25].

Thickness, µm	2
Nano-layer thickness, nm	20–40
Crystal structure	FCC nano-crystalline/laminated
Grain size, nm	20–40
Nano-layer thickness, nm	20–40
Micro-hardness, GPa	30

The micro-hardness of the coating was measured on WC-Co substrates using a Micro Material NanoTest system. Nanoindentation was performed at room temperature in a load-controlled mode with a Berkovich diamond indenter according to an ISO14577-4 procedure.

Cutting data are presented in Table 3.

Table 3. Cutting parameters used for the tool life evaluation.

Machine	**Cutting Parameters**				
Three-axis vertical milling center (Matsuura FX-5).	Speed, m/min	Feed, mm/tooth	axial depth, mm	radial depth, mm	Coolant
	600; 700	0.06	5	0.6	Dry conditions

Cutting tests were performed during dry ball-nose end milling (Mitsubishi carbide ball-nose end mills, D = 10 mm) of the hardened AISI H13 tool steel with hardness HRC 53–55. Cutting parameters are outlined in Table 3. The coated tool flank wear was measured using an optical microscope (Mitutoyo model TM). At least three cutting tests were performed for each type of coating under the corresponding conditions. The scatter of the tool life measurements was approximately 10%.

The structural and phase transformation at the cutting tool/workpiece interface, as well as the chemical composition of the formed tribo-films were determined by a Physical Electronics (PHI) Quantera II (Physical Electronics Inc., Chanhassen, MN, USA) X-ray Photoelectron spectroscope equipped with a hemispherical energy analyzer and an Al anode source for X-ray generation and a quartz crystal monochromator for focusing the generated X-rays. A monochromatic Al K-α X-ray (1486.7 eV) source was operated at 50 W–15 kV. The system base pressure was as low as 1.0×10^{-9} Torr, with an operating pressure that did not exceed 2.0×10^{-8} Torr. Before any spectra were collected from them, the samples were sputter-cleaned for four minutes by a 4 kV Ar+ beam. A pass energy of 280 eV was used to obtain all survey spectra, and a pass energy of 69 eV was used to collect all high-resolution data. All spectra were obtained at a 45″ take-off angle using a dual beam charge compensation system to ensure the neutralization of all samples. The instrument was calibrated using a freshly cleaned Ag reference foil, where the Ag 3d5/2 peak was set to 368 eV. All data analyses were performed using PHI Multipak version 9.4.0.7 software. Areas of high-resolution (HR) HR analysis were selected based on a careful preliminary investigation of general photoelectron spectra of the worn surface close to the buildup edge area.

A cross-sectional TEM analysis combined with FIB (focused ion beam) was used to investigate the worn coating layers on the cemented carbide WC/Co substrates. Transmission electron microscopy was conducted with a JEOL FS2200 microscope (Tokio, Japan) at an acceleration voltage of 200 kV.

3. Results

A microscopic volume seizure (see TEM cross-section image, Figure 1) was observed at the very beginning of cutting (only after 2 min, Figure 1). This is an example of a stick-slip process, which occurs during chip formation and involves a self-organized critical phenomenon [11,12]. The forming adhesive layer of workpiece material gradually transforms into a BUE (buildup edge) [11,12].

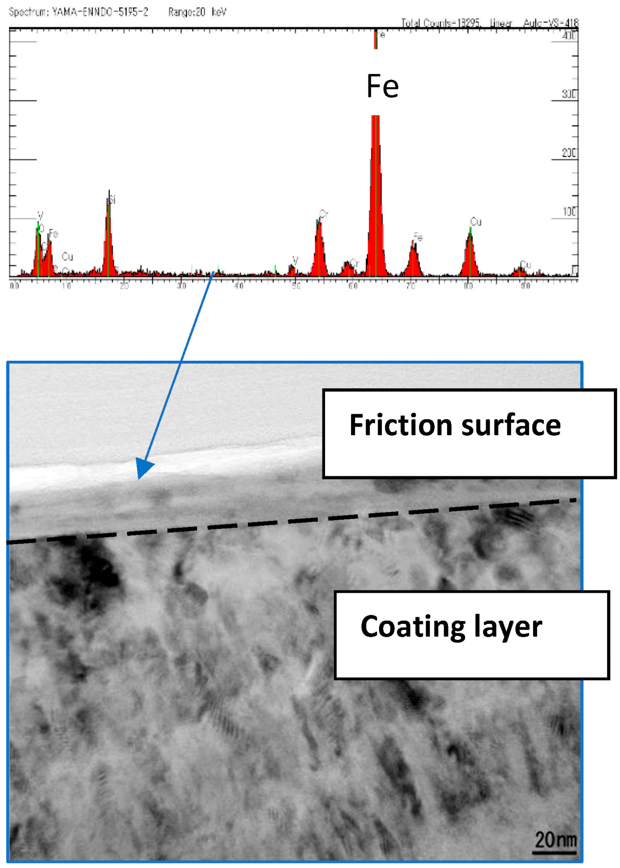


Figure 1. Formation of an initial adhesive layer on the rake surface of the coated cutting tool during the very beginning of wear (length of cut of 2 m). TEM cross-section of the worn surface combined with EDS data.

A strong adaptive response takes place within the layer of the analyzed complex adaptive system represented by a nano-multilayer TiAlCrSiYN/TiAlCrN PVD coating. This is confirmed by the formation of the highest amount of thermal barrier tribo-films at the very beginning of the cutting process (Figure 2a,b).

The coating layer has a strongly non-equilibrium state in the as-deposited conditions [10]. Due to this, a mass transfer of the necessary elements (such as Al, Cr, Si, Y) to the friction surface is accelerated, resulting in the formation of protective tribo-films (Figure 2a,b). This constitutes a self-catalyzing process under harsh tribological conditions. At the same time, the formation of tribo-films itself has certain similarities to autocatalytic chemical reactions in which at least one of the reactants is also a product [4–6]. XPS data presented in Figure 2c confirms this hypothesis. Previously published data indicated that elementary Ti, Al, Cr, Si, and Y tribo-oxides are formed on the friction surface [15]. These tribo-oxides interact in a diverse manner by forming complex tribo-oxides, such as mullite and even garnet (see Figure 2c). All of these oxides, especially the aforementioned two, have very low thermal conductivity [20–22].

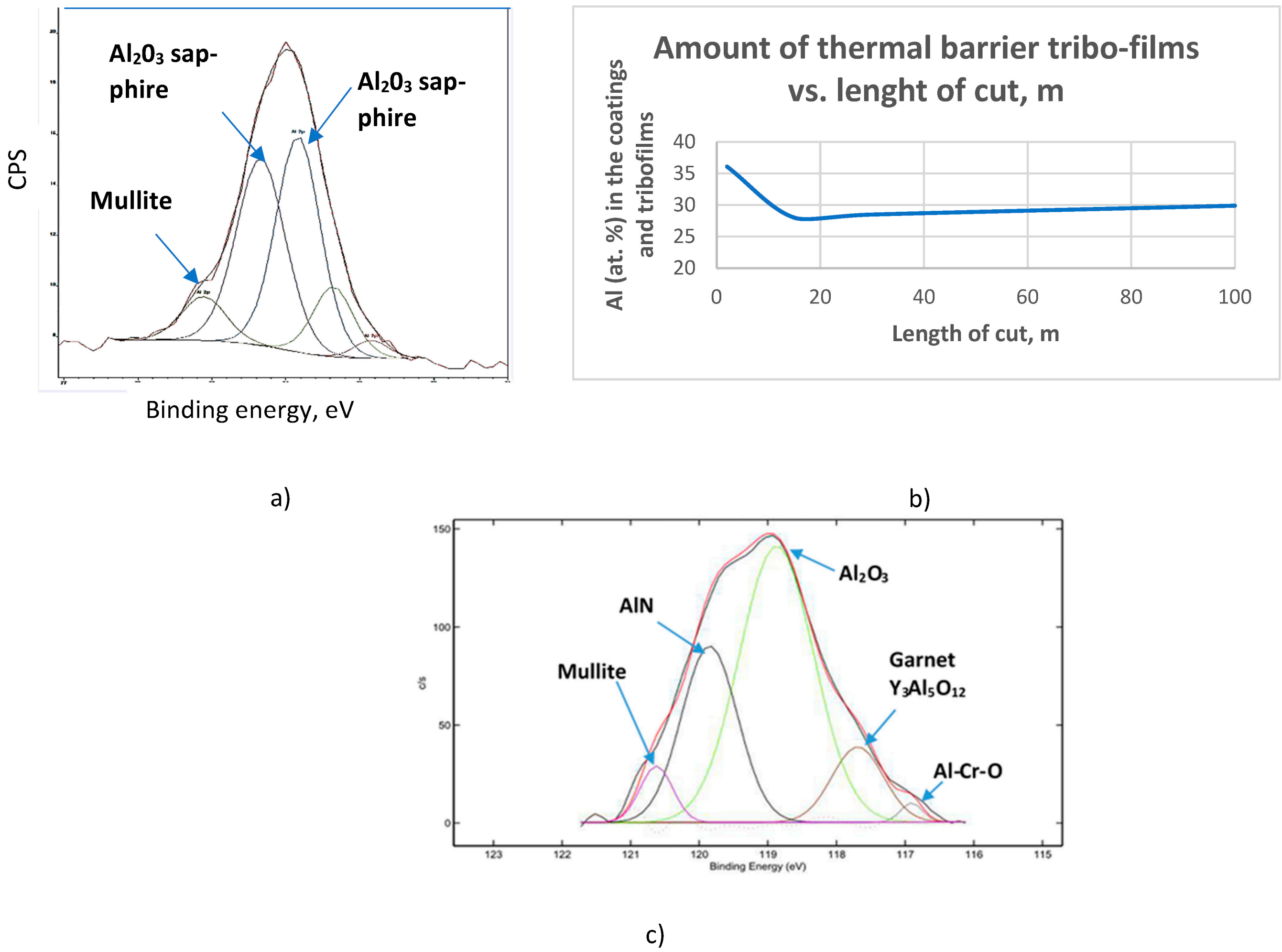


Figure 2. Formation of thermal barrier tribo-films on the friction surface of the coated cutting tool: (**a**) thermal barrier tribo-films formed at the very beginning of cutting (a length of cut of 2 m according to XPS data in the Al2p spectrum); (**b**) amount of thermal barrier tribo-films vs. length of cut (XPS data). A self-catalyzing process that involves the formation of an increased amount of tribo-films at the very beginning of cutting; (**c**) the formation of various complex thermal barrier tribo-films (XPS data; Al2s spectrum), also an auto-catalytic process.

These combined effects could be used to control the wear process in a way that increases tool life. One of the key features of the analyzed adaptive coatings is their ability to deliver improved surface protection under increasingly harsh operating conditions [23]. Due to the elevated operating temperatures at such cutting conditions, the formation of complex protective oxide tribo-films is most intense during the very beginning of the running-in stage of wear (Figure 2). This can help achieve a considerable improvement in tool life (Figure 3).

Figure 3 presents the tool life data of ball-nose end mills with an adaptive TiAlCrSiYN/TiAlCrN multilayer PVD coating under dry machining conditions at speeds of 600 and 700 m/min m/min. The initial short-term acceleration (a length of cut of 5 m) of up to 700 m/min, followed by a deceleration to 600 m/min, is also presented in the graphs. Short-term acceleration during the running-in stage leads to the stabilization of flank wear at the beginning of the cutting process (see insert in Figure 3). At a speed of 600 m/min, flank wear gradually stabilizes (Figure 3). At a speed of 700 m/min, flank wear grows quite fast, which results in a very low tool life. At this extremely high speed, the wear conditions are close to the edge of chaos [8]. During the brief period of acceleration at 700–600 m/min, flank wear values stabilize faster than at 600 m/min, below a length of cut of 5 m (insert in Figure 3). This can be attributed to the accelerated mass transfer of coating elements to the friction surface, which promotes the formation of protective tribo-films (Figure 2). A considerable improvement in tool life can be thus achieved if the cutting speed is accelerated during the running-in stage of wear. The period of speed acceleration needs to be brief to promote the enhanced formation of protective tribo-films, but at the same time, prevent excessive damage of the coating layer due to thermal fatigue [24]. This short-term speed-up was found to have extended tool life by 34% (Figure 3).

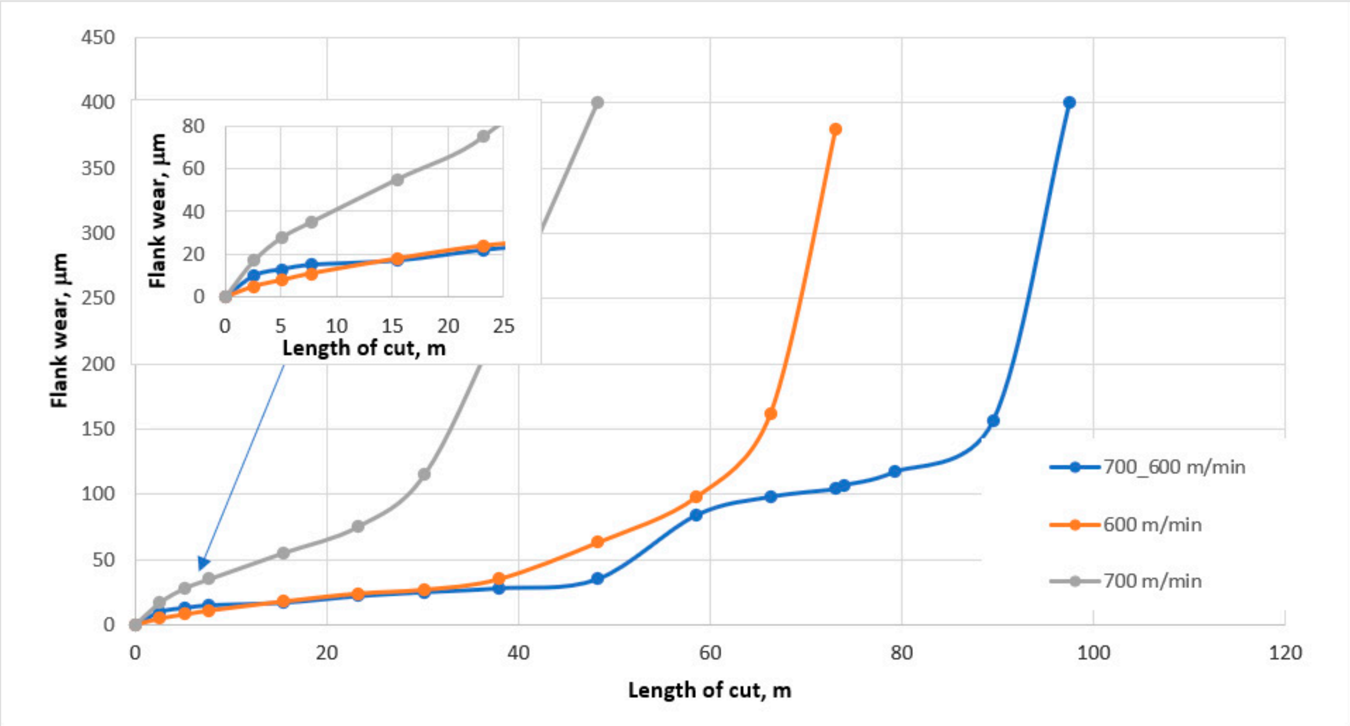


Figure 3. Flank wear at various cutting speeds and with initial short-term acceleration at the very beginning of cutting in tools with a TiAlCrSiYN/TiAlCrN multilayer coating.

Figure 4 shows the relationship between the wear rate and the length of cut.

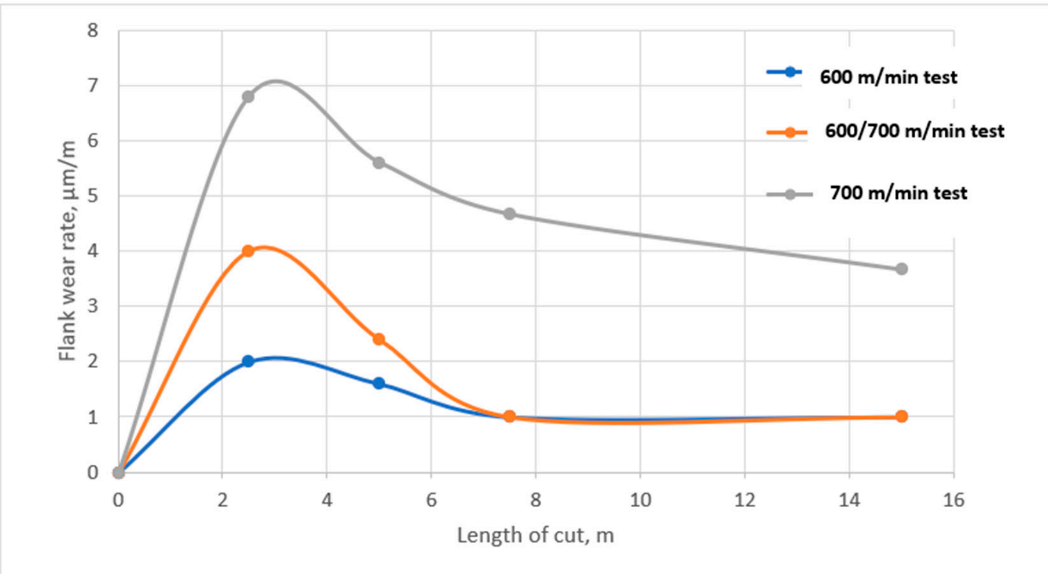


Figure 4. The flank wear rate of tools with a TiAlCrSiYN/TiAlCrN multilayer coating under varying machining conditions.

The relationship between wear intensity and entropy production is shown in [23]. A relatively sharp decrease in the wear intensity in Figure 3 can be attributed to the passage of self-organization [25].

It should be noted that the relationship between wear rate and length of cut must pass through a maximum peak before self-organization can occur (as evidenced by a sharp decrease in the wear intensity). The corresponding relationship between the entropy production and wear rate also follows the same trend.

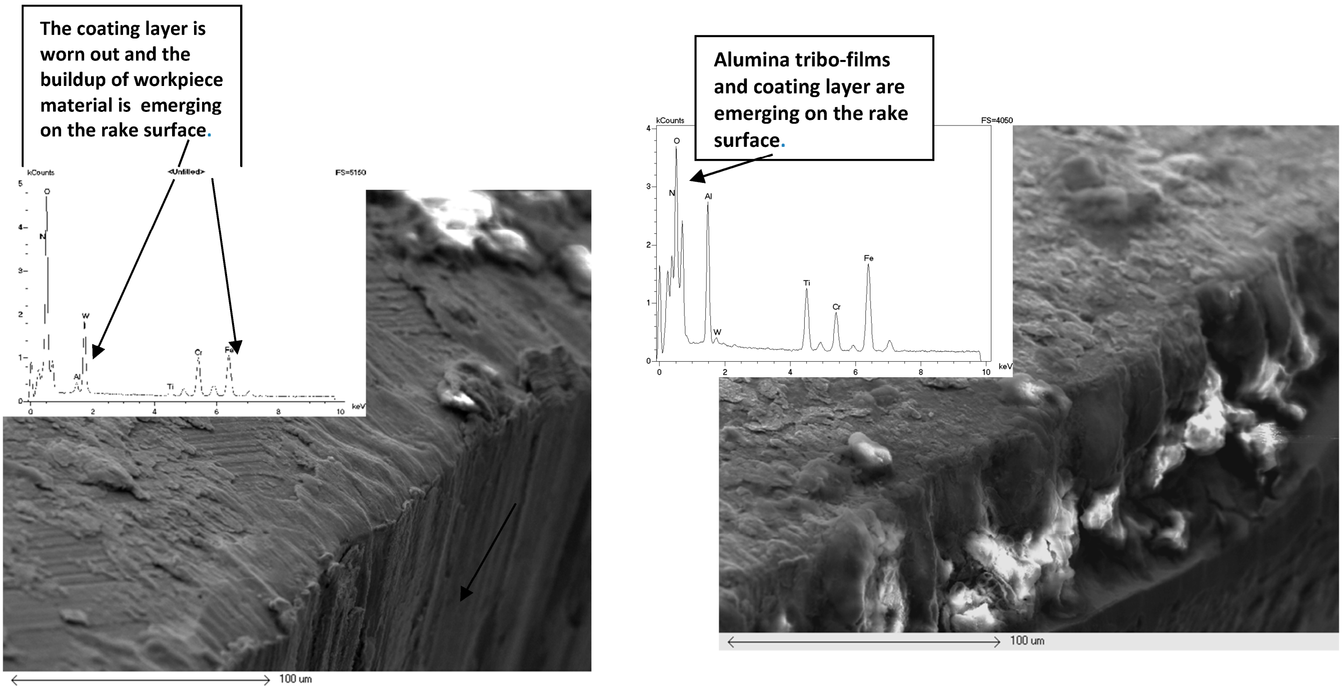
There is a significant difference in curves under the analyzed cutting conditions. At an ultra-high speed of 700 m/min, flank wear and wear rate are extremely high. The wear rate strongly spikes at the beginning before slowing down, although its values are much greater than under the other analyzed cutting conditions (Figure 4). Under the latter cutting conditions, wear intensity also increases during the beginning stage of cutting, before being reduced to a lower range. At 600–700 m/min, the wear rate spike is much greater than in the 600/min test. This is due to the enhanced formation of thermal barrier tribo-films. It should be noted that the analyzed phenomena involve a SOC process. At the higher temperature generated during the initial speed-up, this SOC (consisting of an adhesive layer and BUE formation) is more intense. As shown in [12], the resulting growth of entropy production immediately prompts an adaptive response from the surface-engineered layer to external stimuli, which directly intensifies energy dissipation. Thus, the probability of the occurrence of self-organization grows with increased entropy production (energy dissipation) [24].

Temperature gradients, mechanical stresses, and chemical potentials all rise along with cutting speed. An increase in gradients (thermodynamic forces) leads to the intensification of heat flows, material transfer under the influence of mechanical stresses, diffusion, and chemical reactions (thermodynamic flows). This situation increases the likelihood of self-organization, while at the same time, promoting a significant increase in the wear rate. Dissipative structures are formed during self-organization, characterized by an intensive run of processes with negative entropy production. This leads to a decrease in the wear rate, as can be seen in Figures 3 and 4. If self-organization does not occur at this point, then the wear rate remains high and the tribo-system quickly fails.

Self-organization is one of the major mechanisms of adaptation that form protective layers adapted to function ideally under their incipient conditions (in our case, at a specific cutting speed). Protective layers formed at a cutting speed of 600 m/min will effectively resist wear at this speed, but will less effectively resist wear at a speed of 700 m/min. Accordingly, protective layers formed at a speed of 700 m/min will resist wear at a cutting speed of 700 m/min, but will be even more effective at a speed of 600 m/min. This is because the layers were formed under relatively severe conditions (700 m/min.) and, accordingly, adapted to work at this speed. However, in the future, these layers are working under relatively less severe conditions (speed 600 m/min). Respectively, under less severe conditions, these layers will better resist wear, as can be seen in Figures 3 and 4.

SEM/EDS investigations of end-mill worn patterns show the following (Figure 5).

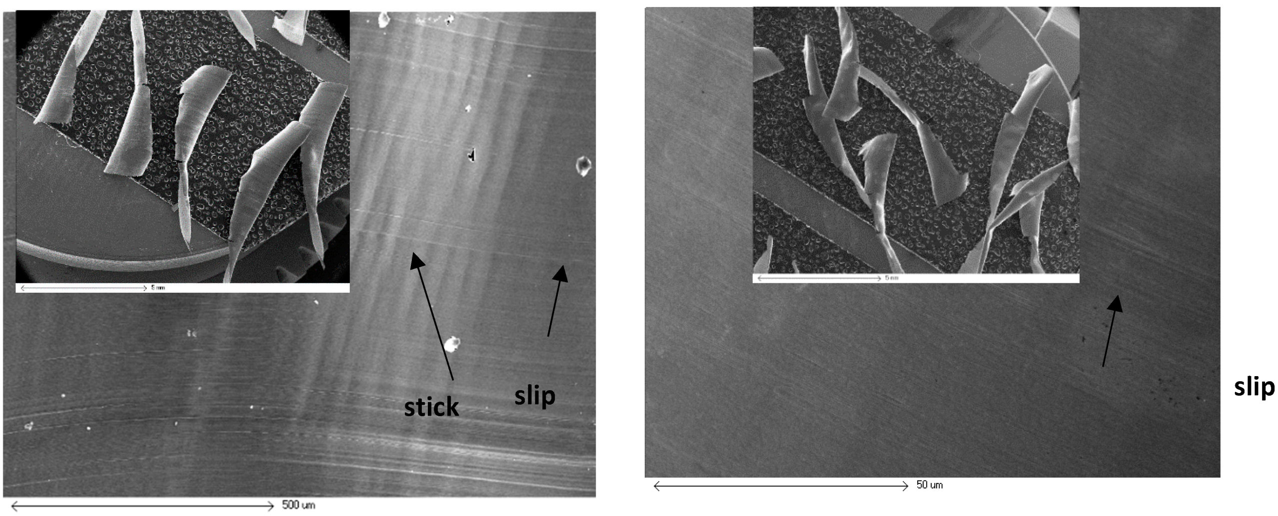
The surface morphology of coated ball-nose end mills tested until the end of tool life at a cutting speed of 600 m/min indicated that the coating had been entirely worn out from the rake surface of the cutting tool. The substrate material (cemented carbide) was exposed, and a buildup of the workpiece material was present (Figure 5a). In contrast, the surface morphology of the coated ball-nose end mill tested until the end of tool life, in which the cutting speed was initially accelerated to 700 m/min and then reduced to 600 m/min, indicated that the coating was still present on the rake surface of the tool (Figure 5b). The formed alumina tribo-films (according to the EDS spectrum in Figure 5b) effectively protected the surface, leading to an overall tool life increase.



(**a**) (**b**)

Figure 5. SEM/EDS images of worn surfaces at the end of tool life (flank wear of 400 microns): (**a**) At 600 m/min, the coating is worn out; (**b**) At 700/600 m/min, the coating with a tribo-oxidized top layer is present on the rake surface.

The improvement of frictional conditions during the short-time speed-up can be also confirmed by chip analysis (Figure 6). The chips formed at the 600 m/min test are less curly and have a rougher undersurface with stick-slip patterns (Figure 5a). At the same time, the chips formed at the 700–600 test are curlier and have a smooth undersurface (Figure 5b). This confirms the formation of beneficial tribo-films, which results in superior surface protection and improved tribological conditions.



(**a**) (**b**)

Figure 6. Chip undersurface formed by tools with a TiAlCrSiYN/TiAlCrN multilayer coating under varying machining conditions: (**a**) a speed of 600 m/min; (**b**) a speed of 700/600 m/min. SEM data.

4. Discussion

The presented data show that the rate of physical–chemical processes is strongly accelerated under the extreme tribological conditions of ultra-high-performance machining at the edge of chaos. At the beginning of wear, during the very initial running-in stage (see Figures 4 and 5), the rise of intensive thermomechanical processes is dependent on intensive adhesive interactions at the tool/chip interface (Figure 1). This is due to the stick-slip phenomenon (Figure 6a) typical of chip formation, itself representing a self-organized critical process [26,27]. The resulting growth of entropy production [28–34] immediately prompts an adaptive response from the surface-engineered layer to the external stimuli, which leads to the intensification of energy dissipation (self-organization) [16]. During the initial stage of friction, tribo-films are generated at a very rapid rate (Figure 2b). A similar trend was already described in [9]. This is the so-called "trigger effect" [15] and is associated with a self-catalyzing reaction. At the same time, an autocatalytic reaction occurs alongside the formation of complex tribo-oxides, such as mullites and garnets, whose thermal conductivity is considerably less than that of elemental oxides, such as sapphires [15] (Figure 2). These reactions are fundamentally nonlinear, which results in a decrease in entropy production (generation of order) as well as wear rate.

It was shown in [4–6] that the necessary tribo-films have been formed on the layer of the end mill's coating after just a few meters of cutting. Moreover, the composition of tribo-films does not change significantly during further cutting. If the thickness of the tribo-films remains constant, then within the stationary state of cutting tools' wear, the rate of the tribo-films recovery corresponds to the wear rate of the tribo-system. We can consider this as an element of autocatalysis, when an increase in the wear rate leads to an increase in the recovery rate of tribo-films. In (Ti-Al)N-based coatings, a relatively low wear rate of the tool is caused by the predominant formation of aluminum oxide (Al_2O_3) tribo-films on the friction surface. With the predominant formation of titanium oxide (TiO_2), intense tool/workpiece adhesive interaction occurs, which leads to tool wear [11]. As a result of this situation, mainly titanium oxide wears out. This leads to the formation of an increased amount of aluminum oxide tribo-films and correspondingly to a wear rate decrease. This can be an example of a kind of autocatalysis, when tribo-films that have a reduced wear rate are formed during the interaction of tribo-films. The materials that interact during the friction process (tribo-films) are a result of the friction process (complex tribo-films, which have a reduced wear rate). The present study is concerned with the wear performance of TiAlCrSiYN/TiAlCrN multilayer PVD coatings. These coatings have a high enough number of alloying elements in their composition (5 metallic elements) and can operate at very high temperatures (above 1000 °C). Therefore, the probabilities of either self-catalyzing or autocatalyzing processes occurring are very high, which correspondingly leads to the acceleration of self-organization-based processes. All these result in tool life improvement.

5. Conclusions

Physio-chemical processes corresponding to various catalyzing reactions (self-catalyzing and autocatalyzing) were observed in the complex adaptive surface-engineered system represented by nano-multilayer TiAlCrSiYN/TiAlCrN PVD coating under extreme tribological conditions of ultra-high-performance machining. The rate at which the sequence of events takes place on the friction surface is strongly accelerated at the very beginning of cutting. Experimental studies showed that accelerated tribo-film formation had resulted from a combination of self-organization and self-organized criticality phenomena under the outlined tribological conditions close to the edge of chaos. A means of control over this process is presented as well, achieved by the optimization of machining conditions (an initial short-term increase in the cutting speed during the very beginning of the running-in stage) with the goal of improving the wear performance of the coated cutting tools.

Author Contributions: G.S.F.-R.—conceptualization, writing—original draft preparation; I.S.G.—writing, review and editing; J.L.E.—draft editing. All authors have read and agreed to the published version of the manuscript.

Funding: J.L.E. gratefully acknowledges funding from Spanish Ministry of Science (Projects PID2021-128727OB-I00 and TED2021-132752B-I00).

Data Availability Statement: Data unavailable publicly.

Acknowledgments: Critical proofreading of the manuscript by Michael Dosbaev is acknowledged.

Conflicts of Interest: The authors declare no conflict of interest.

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