



# TOPOCROM – Rethinking the fundamentals of electrocrystallisation

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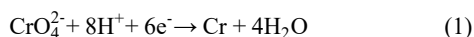
## Abstract

This article describes the scientific electrochemical and material-technical background of a technology with which hard chrome layers can be specifically provided with a topography, i.e. with a microstructure. The structure is characterised by the formation of spherical cap-shaped structural elements. This process, called TOPOCROM®, has been successfully available on the market for some time and is applied exclusively by a German job plater. As is often the case in the electroplating industry, which is characterised by medium-sized companies, the original development was based on the trial and error principle. With increasing industrial success, the need to better understand the basic mechanisms grew, which led to the investigations presented in this paper. First, theoretical considerations are made based on the fundamentals of electrocrystallisation. These lead to the assumption that a change in the crystallisation form occurs when the structures are generated by the so-called current ramp. This assumption was verified by experiments in which flat discs were plated and then measured using EBSD. This proved that the suspected change in electrocrystallisation actually takes place, more precisely from a strongly field-oriented {111} texturing to an unoriented crystal form. On this basis, a growth model for the special layers was then developed.

## 1. Introduction

### 1.1 Traditional chromium plating

Chromium plating itself has a long history. In 1797, L.N. Vauquelin succeeded for the first time in producing metallic chromium from red lead ore,  $\text{PbCrO}_4$ , which was discovered by J.G. Lehmann in 1766 [1]. It was not until the middle of the 19<sup>th</sup> century that the development of electroplated chromium deposition solutions became the focus of attention. Chrome-plated workpieces are considerably more resistant to corrosive media and mechanical wear than non-plated or nickel-plated workpieces and have a decorative, bluish metallic lustre. Chromium is mostly deposited from chromate ions (Equation (1)) [2].



Due to the significantly more demanding electrolyte management of trivalent electrolytes compared to hexavalent electrolytes, it crystallised in the early 20<sup>th</sup> century that deposition from hexavalent chrome electrolytes would prevail over CrIII electrolytes, which was demonstrated by F. Salzer but above all by E. Liebreich in a patent [3,4]. As a result, developments from then on focussed on deposition from hexavalent electrolytes.

A basic distinction can be made between two areas of chrome plating, even if these cannot be completely separated from each other.

On the one hand, this is bright chrome plating or decorative chrome plating. Bright chrome plating is when the deposited chrome layer

thickness is between 0.1  $\mu\text{m}$  and 1.5  $\mu\text{m}$ . In recent years, hexavalent electrolytes have been increasingly replaced by trivalent electrolytes as a result of the European chemicals regulation REACH. This coating is primarily used in decorative applications such as in the fittings, automotive and furniture industries. Here, particular value is placed on the high lustre and bluish appearance of the chrome layer. Coating systems are usually used for these areas of application, as a thin layer of chrome offers hardly any corrosion protection and no lustre-forming influences. For this reason, the components are nickel-plated before chrome-plating [5]. As the chrome layer reflects the shine of the underlying layer, different looks can be created using bright nickel layers or velour nickel layers.

Hard chrome plating, on the other hand, is, in simple terms, when the layer thicknesses are so thick that the chrome layer can bring its mechanical properties to bear. From a metallurgical point of view, bright chrome and hard chrome are largely the same apart from the thickness of the layer.

The term hard chrome plating is therefore used when the deposited chrome layers are more than 1  $\mu\text{m}$  thick. The layers deposited in this way are ideally suited as final layers for components that are subject to high wear or high mechanical stress, for example for plating pressure cylinders, feed rollers or piston rods [1]. The layer thicknesses can range from a few  $\mu\text{m}$  to several mm.

Hardness values of 800 HV to 1100 HV are given in the literature, in practice values between 600 HV and 1200 HV can be measured [6], whereby coatings below a hardness of 900 HV are not used, as they do not have the optically high-quality appearance of harder chrome coatings.

It should be noted at this point that all chrome layers are deposited smoothly, or at least in such a way that they reflect the structure of the substrate and do not form their own structure.

On the other hand, there have long been various technical applications in which the wear and corrosion resistance of electroplated hard chrome layers is combined with a topography/microstructure, e.g.:

- Moisture driver cylinders in printing machines are equipped with structured chrome layers so that the chrome layer, which is difficult to wet, can transport the printing medium to moisten the actual printing plate.
- In cold rolling mills for steel, the last rolls, known as skin pass rolls, are equipped with a structured chrome layer that transfers its topography as a negative imprint into the steel sheet [7]. Without this microstructure in the steel sheet, it would not be formable because the forming aiding agent could not be retained and it would also not be possible to paint it with a good result.
- Thread-guiding elements in textile, glass fibre or carbon fibre production must also be microstructured and chrome-plated, as the fibres are very abrasive and would tend to stick to a high-gloss chrome surface, resulting in fibre breakage.
- Feed rollers in straightening cassettes in the sheet metal industry to create targeted grip. Previous technologies for producing structured chrome layers are based on two decoupled processes.

In most cases, the structuring is carried out first and then the structured steel surface is chrome-plated. Less commonly, the already deposited chrome surface is structured. The structuring itself is carried out by sandblasting, electron beam or laser treatment. [8]. From the point of view of efficient production technology, a technology that allows structuring and wear-resistant plating to be carried out in a single process step would be very advantageous. This led to the need to develop a technology that would allow these two previously separate process steps to be carried out in a single step. In addition, there was a requirement for this to be highly reproducible and to allow for precise adjustment of the roughness parameters and topography. The key to solving this problem was an 'it's not a bug, it's a feature' approach, which required a deep understanding of the effects of electrocrystallisation and led to the development of TOPOCROM®.

## 1.2 Introduction of TOPOCROM®

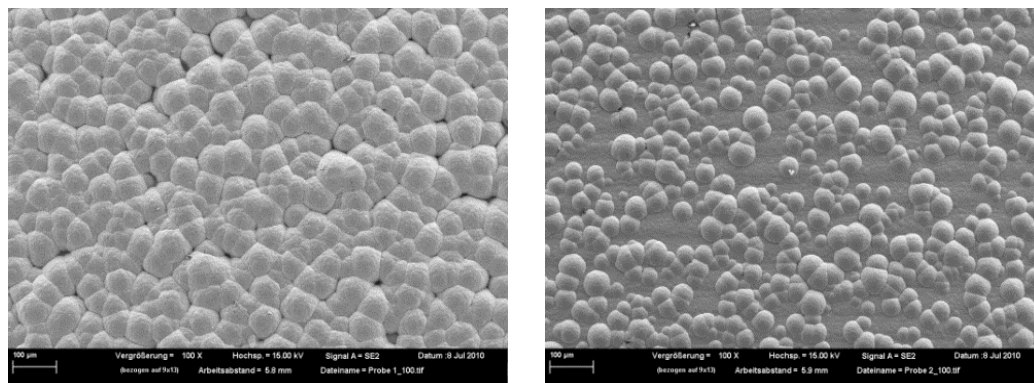
It has long been known in research and industrial practice that electroplated layers, especially chrome layers, become rough when

the usual range of process parameters, particularly with regard to current density and electrolyte temperature, is exceeded. In practice, this is referred to as 'burning'. The basic idea was to control and reproduce this burning in order to develop a production technique that combines structuring and coating. This technology, now known as TOPOCROM, has shown that burning and the resulting roughening can be specifically controlled by significantly increasing the electro-deposition potential and thus the cathodic current density in the form of a staircase function beyond the usual level and at the same time working at a reduced electrolyte temperature. Extensive testing has yielded knowledge on how the formation of the structural elements can be reproducibly controlled by adjusting the variables within the staircase function (step height and depth as well as maximum current density). At the same time, however, it has also been shown that the structural layer must be post-chromed using standard process parameters, as the structural layer itself has proven to be soft and not very wear-resistant. However, since all process steps are carried out in one and the same reactor, plating and structuring are a single process. From a scientific point of view, this raised the question of the previously unknown deposition mechanism. The considerations and experimental evidence are discussed below.

Figure 1 shows an example of TOPOCROM structures in the top view in the SEM: on the left a so-called closed structure (all structural elements grow into each other), on the right an open structure (the structural elements do not touch each other in the majority).

Figure 2 shows a diagram explaining the staircase function mentioned above. It should be emphasised that, for reasons of control technology in connection with the maximum possible currents (up to 40,000 A, depending on the component size), the control is not carried out via current increases but via voltage increases, which result in the corresponding increases in current densities. The control variables are the voltage increase per step (called  $\Delta u$ ), the waiting time between voltage increases (called  $\Delta t$ ), the final current density of the ramp and the holding time of the final current density.

The choice of individual parameters depends on the target topography. Details cannot be provided here, as this is subject to strict confidentiality within the company. Typical values used in practice are  $\Delta u$  0.05 V to 0.5 V with a final voltage of 12 V required to achieve the final current density, which means 240 step to 24 step. Typical values for  $\Delta t$  are 0.5 s to 10 s. The final current density used is 80 to 100 A·dm<sup>-2</sup>, which is maintained for a period of 10 s to 200 s.



**Figure 1.** Closed structure on the left, open structure on the right

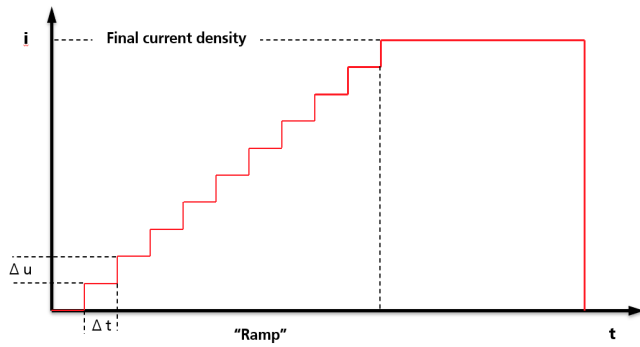


Figure 2: diagram of the staircase function

The change in electrolyte temperatures from pre-chrome (50°C) to structural chrome (32°C to 35°C) to final chrome (50°C) cannot be achieved by heating and cooling the electrolyte volumes used in practice.

In the reactor systems used, the electrolytes are drained and the electrolyte of the other temperature is pumped in as quickly as possible. It is important to keep the parts constantly moist by means of spray rings installed in the reactors, as otherwise adhesion problems will arise.

## 2. Fundamental considerations

The most fundamental and comprehensive treatise on electrocrystallisation effects in metallic deposition, which is still valid today, was written by Prof. Hellmuth Fischer in 1954. 717 pages are devoted to the book title "Electrolytic Deposition and Electrocristallisation of Metals" [9]. In essence, four basic types of electrocrystallisation are derived and defined. The respective metal always crystallises in the crystal lattice that is characteristic of it, in the case of chromium as a body-centred cubic lattice with a lattice constant of 2.9232 Å.

The defined crystal forms by Prof. Fischer are:

The field-orientated texture type, referred to below as FT. Here, individual crystal columns grow in isolation from each other. This form does not form a compact layer and is therefore not used in electroplating practice.

The base-orientated reproduction type, referred to below as BR. Here, compact layers grow in such a way that relatively large crystals are formed that continue the crystal structure of the base material. This can be easily visualised, for example, when copper layers are deposited on copper.

The field-orientated texture type, referred to below as FT, in which compact layers are formed from crystal columns growing together. This form is very common in electroplating. The preferred orientation of the crystals within the columns depends on the deposited metal and the process parameters.

The unorientated dispersion type, referred to below as UD. This forms a compact layer and this consists of crystals lying randomly on top of each other, as if a wheelbarrow of cubes were being poured out.

For the following discussion, it should be noted that there is a consensus in the literature and in practice that hard chromium layers crystallise in the FT with a strong {111} texture, which means that the cube-shaped crystals in the columns stand on their tip.

If we summarise Hellmuth Fischer's explanations and simplify them with regard to the conditions that lead to the transition from one crystal form to another, the picture shown in Figure 3 emerges.

In metallographic examinations of the TOPOCROM layers in cross-section, strong etching with an etchant developed by the Fraunhofer IPA's electroplating department itself at 10 min etching time and room temperature revealed a conspicuous feature when viewed with the SEM, which can be seen in Figure 4.

Below and above the actual structure-forming zone, a metallographic image can be seen that is to be expected from hard chrome layers under these preparation conditions. It should be noted that, as already mentioned above, the structure layer is covered with a conventional hard chrome layer and that normal hard chrome plating parameters are also present at the start of the structure-forming current ramp. This makes the etching pattern above and below the structure-forming zone, easy to explain. What is striking, however, is the sharply defined and completely different-looking image within the structuring zone. It should be taken into account that the structuring phase takes place with the same chrome electrolyte but at a lower electrolyte temperature (hard chrome plating 55°C, structure phase 35°C) and that current densities are achieved within the structure-forming ramp that are significantly higher than those of normal hard chrome plating (hard chrome 50 A·dm<sup>-2</sup>, structure ramp up to 80 A·dm<sup>-2</sup>).

If all these facts are taken into account and brought into line with the systematics of electrocrystallisation shown in Figure 2, the assumption arises that electrocrystallisation changes from the FT to the UD type during the structuring phase (lower electrolyte temperature with simultaneously increased current density). The proof of this is discussed in the following paragraph.

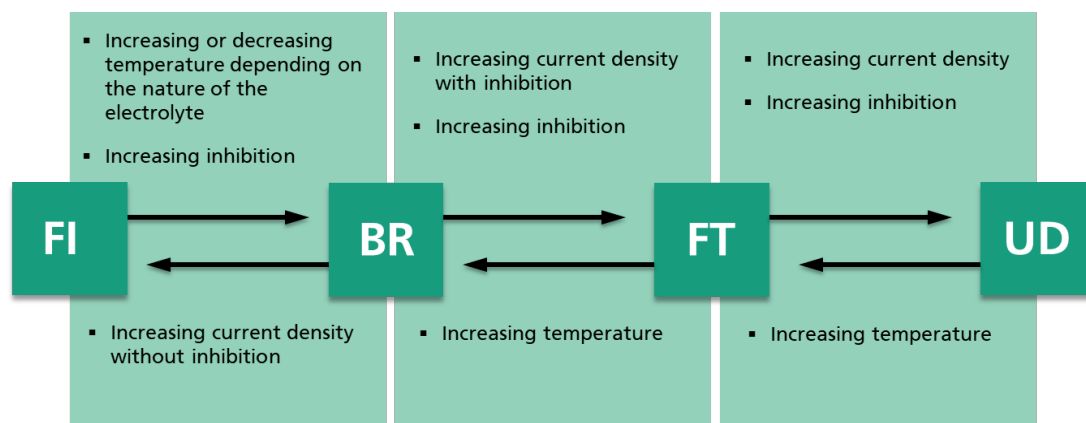
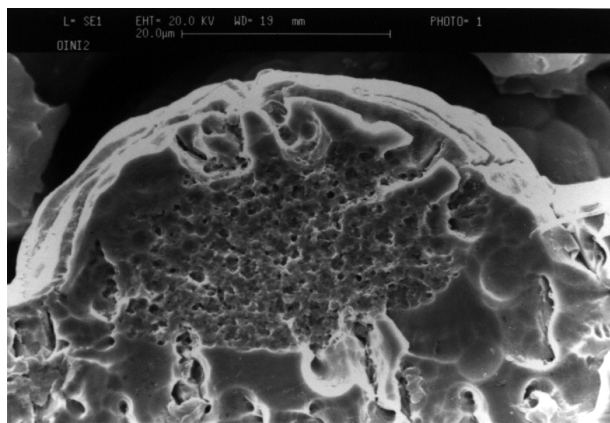


Figure 3. Classification of the transition between the crystal forms according to Hellmuth Fischer [9]



**Figure 4.** Heavily etched TOPCROM structural element in cross-section under a scanning electron microscope.



**Figure 5.** Experimental device.

### 3. Experimental proof of the mechanistic considerations

Firstly, consideration was given to which measuring method could be used to provide the desired evidence. Crystal textures can be

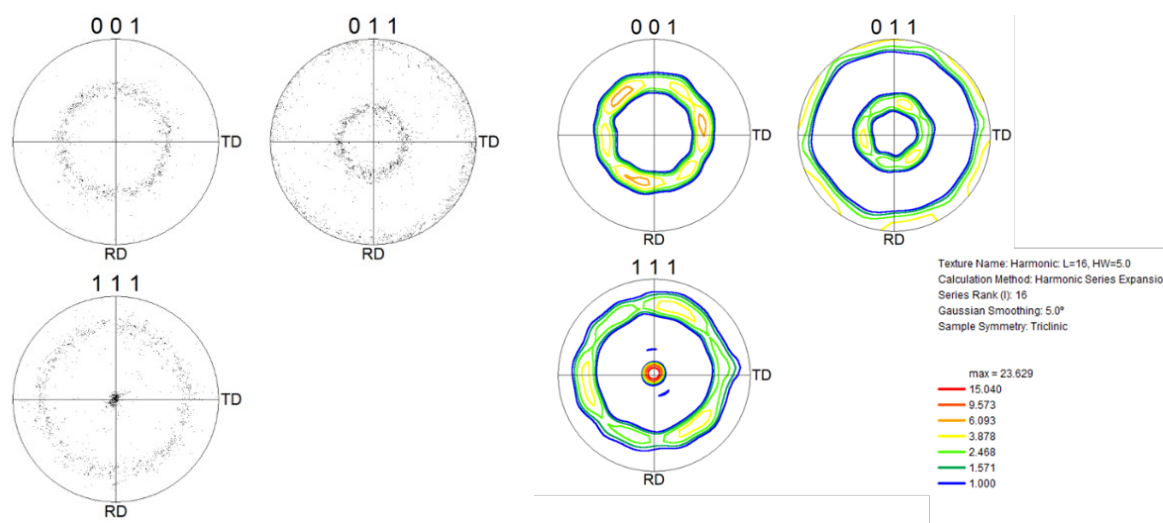
analysed with comparable significance using X-ray diffractometry (XRD) or electron backscatter diffraction (EBSD). EBSD was chosen as the method due to its availability.

The samples were then defined: for a reliable and meaningful measurement, they had to be flat and naturally very evenly lated with a surface area of at least 20 mm × 20 mm. Steel blanks measuring 40 mm × 40 mm and 3 mm thick, made of 42CrMo<sub>4</sub> steel, ground and polished to a roughness value of Rz 3 µm, were chosen. Before chrome plating, the test specimens were coated with 20 µm high-gloss nickel from a Watts nickel electrolyte at a current density of 5 A·dm<sup>-2</sup> and a temperature of 50°C. In order to ensure uniform conditions, a basic chrome plating of 10 µm at a current density of 50 A·dm<sup>-2</sup> and an electrolyte temperature of 55°C was also carried out. The chrome electrolyte used for this was a standard chrome electrolyte with 250 g·L<sup>-1</sup> CrO<sub>3</sub>, 2.5 g·L<sup>-1</sup> H<sub>2</sub>SO<sub>4</sub> and 5 g·L<sup>-1</sup> Cr<sup>3+</sup>. The same electrolyte was then used to apply the actual test layers.

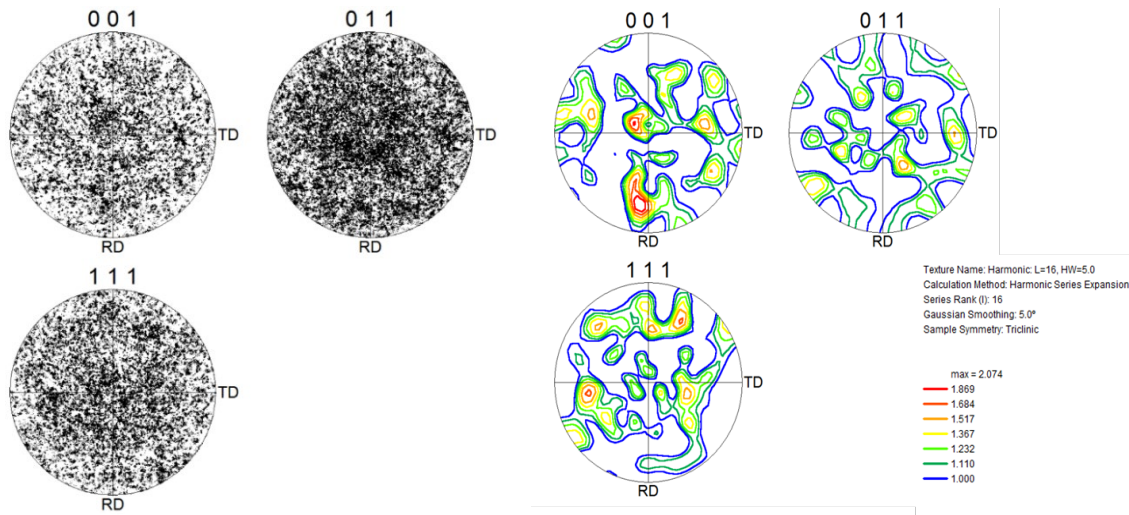
Since the aim was to deposit the chromium layers as uniformly as possible and this is dependent on the primary electric field distribution, a special device was manufactured that was designed using electrochemical field simulation (using COMSOL Multiphysics) and ensures a homogeneous distribution of the electric field on the samples and thus a homogeneous plating by covering the edges. Figure 5 shows this device, on the left in the drawing installed in a 2 L beaker with a round anode as a counter-anode, on the right in the assembled practical state with a built-in round anode which was also used to run the simulation.

The electrolyte already mentioned above was used for the pre-chromium plating and the chromium plating experiments themselves. A specially manufactured double-walled 2 L beaker was used; the double wall served to cool the electrolyte. A JULABO laboratory cooler type FL 601 with corresponding control was used as the cooler, and a Plating Electronic laboratory rectifier POWER STATION pe1018 was used as the rectifier. The beaker was positioned on an IKA laboratory magnetic stirrer IKA C-MAG HS 7 IKAMAG<sup>®</sup> and stirred at a constant speed.

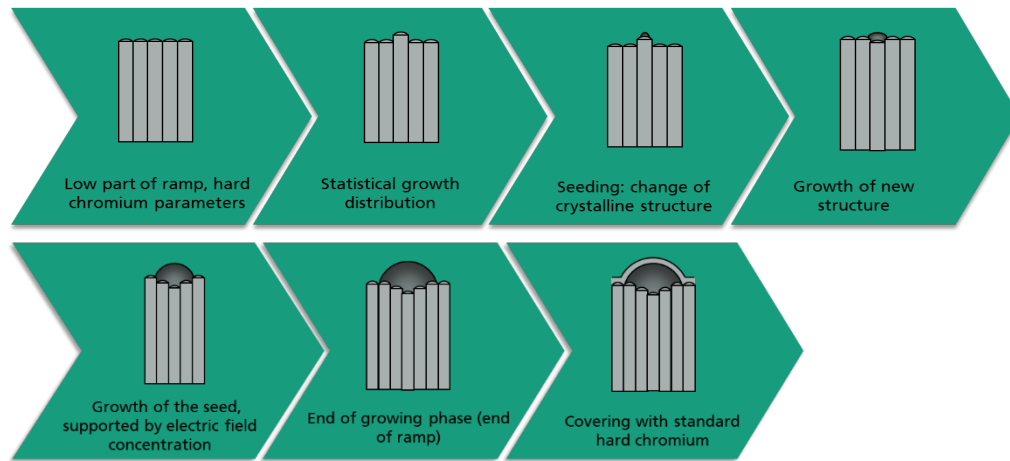
For reasons of reproducibility, three tests were carried out in each case and the results were absolutely identical. Standard hard chrome samples coated at 50 A·dm<sup>-2</sup> and 55°C for 20 min served as reference samples (samples 1).



**Figure 6.** Pole figures from EBSD- Scan Sample 1.1 (left), Calculated Pole figures from EBSD- Scan (from average orientation per grain) Sample 1.1 (right).



**Figure 7.** Pole figures from EBSD- Scan Sample 2.1 (left), Calculated Pole figures from EBSD- Scan (from average orientation per grain) Sample 2.1 (right).



**Figure 8.** TOPCROM growth model.

For the test samples (sample 2), parameters were used that correspond to the upper part of a TOPCROM structuring ramp and were not subsequently further chromium-plated, as the EBSD measurement has a very low penetration depth. This made it possible to ensure that the crystal structures that are also present inside a structure-forming element are created. After pre-chromium plating, these samples were coated with a current density of  $80 \text{ A} \cdot \text{dm}^{-2}$  at a temperature of  $35^\circ\text{C}$  for 10 min. Since the current density was not increased in the form of a staircase function, only a negligible roughness was produced. The significantly shorter coating time of samples 2 compared to samples 1 can be explained by the fact that the current density is higher and the Faraday current yield is also higher due to the applied process parameters.

#### 4. EBSD examination results

The following diagrams show the results of the EBSD measurement carried out.

As was to be expected with the hard chromium parameters, these pole figures show a clear  $\{111\}$  texture. This proves that sample production, preparation and evaluation deliver correct results. The EBSD measurements on samples 2 are completely different.

These measurements clearly show that cubic body-centred crystals are present, but that they have grown without any significant preferential orientation. The idea derived from the fundamentals that a change to the UD type according to Fischer takes place during the growth of the TOPOCROM layer is therefore clearly proven.

Based on these results, a growth model can be derived, which is illustrated in Figure 8 below. The chromium layer grows at the beginning of the current ramp (staircase function) with still moderate current densities as a normal hard chromium layer with a  $\{111\}$  texture. While the current density is increased in steps, the crystal columns grow next to each other, with some of them slightly ahead of others for statistical reasons. At a point determined by other parameters (electrolyte temperature and composition), crystallisation changes to the UD type; the number of points at which this happens and therefore the distribution density of the structural elements depends on the height of the steps. The electric field lines concentrate microscopically on these now set nuclei, while the current density is further increased. This explains the spherical cap-shaped growth. How long the chromium layer grows under these parameters determines the size of the structural elements and thus the roughness of the layers. As the chrome layer in UD type is relatively soft (values of approx.  $650 \text{ HV } 0.05 / \text{Test load}$

0.49 N, applied for a period of 15 s in a Zeiss Axio Lab.A1/ were measured), the structures are plated with a normal hard chrome layer. All this takes place in closed, emission-free and REACH-compliant plants specially developed for this coating. The changeover between electrolytes of different temperatures (but the same composition) is seamless, so that structuring and coating undoubtedly take place in one process.

## 5. Summary

Based on fundamental mechanistic considerations, a model of the electrocrystallisation of a very special and innovative chrome plating technology was established, which was verified by highly reproducible experimental investigations and measurements. A growth model of this unique plating was derived from this. With this knowledge, structures can be adjusted even more precisely. Future work will focus on further fields of application for this type of plating. Work will also begin on how such layers can possibly be deposited from trivalent chrome electrolytes in the future.

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