

## **An Innovative Approach to New Hybrid Coatings based on HiPIMS Technology: The HI3 process**

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Keyword: Arc, sputtering; HIPAC, energy, roughness, hybrid, HiPIMS, advanced coatings

### **Abstract**

A new class of advanced PVD-coaters, the METAPLAS-DOMINO series, for dedicated coating applications comprise both improved vacuum arc evaporators (APA, Advanced Plasma Assisted) and HiPIMS magnetron sputtering sources (High Power Impulse Magnetron Sputtering) traded by SM as HIPAC (High Ionized Plasma Assisted Coating) [1]. The ion cleaning is based on the AEGD process (Arc Enhanced Glow Discharge). This combination of the three highly ionized processes is named HI3 (High Ionization triple = 3). It is possible to run the processes in different modes, e.g. pure APA arc evaporation or pure HIPAC magnetron sputtering. However the combination of the two high ionized deposition processes to generate multilayer, nano-multilayers and nanocomposite layers opens new horizons in tailoring of coating.

The arc evaporation itself is limited to specific cathode material properties (mostly metal alloys). HIPAC magnetron sputtering processes can be used to atomize and ionize materials which are difficult to evaporate or not evaporable by cathodic arc, e.g. Si, SiC, WC, TiB<sub>2</sub> and others. Specific features of the PVD system equipped with APA arc evaporators and HIPAC magnetron sources will be shown. First results of advanced hybrid coatings deposited by the HI3 process will be presented.

### **1. Introduction**

The vacuum arc evaporation and the magnetron sputtering are the PVD deposition methods of Sulzer Metaplas to produce tribological and other functional coatings. The application of vacuum arc evaporation is the dominating PVD method for tool coatings and high performance component coatings. Besides advanced hard coatings also upgraded arc evaporators like APA (Advanced Plasma Assisted) are available.

Classical DC-magnetron sputtering (DC-MS) is mainly used for a-C:H:Me coatings or for the deposition of interlayers for a-C:H:X coatings. Although the DC-magnetron sputtering process often results in smoother coatings the number of coating systems in industrial use for wear reduction of tools is much lower than that of the arc evaporation. The reasons for that are the advantages (stability, productivity, costs, and coating properties) of the arc evaporation process in industrial scale.

#### **Brief description of the arc evaporation deposition process**

The vacuum arc evaporation is a self-sustaining discharge under vacuum condition. The arc process starts with the striking of a high current, low voltage arc on the surface of a cathode. The current density within a cathode spot (diameter ca. 10 µm) goes up to 10<sup>7</sup> A/cm<sup>2</sup> assuming a minimal spot current of 10 A [2]. The temperature at the cathode spot is extremely high resulting in a jet of vaporized cathode material. The localized cathode spot exists only for a short period of time. The spots jump to another area. The plasma jet consists of multiple charged ions, neutral atoms and macro-particles (droplets). A high level of ionization depending on the cathode material (30-100%) is observed [3]. Reactive gases are used to deposit nitrides, carbonitrides, oxynitrides and oxides. The reactivity is also given at low temperature due to excitation, dissociation and ionization. To eliminate the emitted droplets different filter systems are used [4].

### Brief description of the magnetron sputtering deposition process

The DC magnetron sputtering (DC-MS) is based on the atomization of the target material by an energetic bombardment of its solid surface by ions. The sputtering process is possible in either under vacuum condition, or in inert gas or in reactive (inert plus reactive) gas atmospheres. The electrical conductivity of the target material has to be sufficient high. The ionization is mostly dominated by the sputter gas ionization (mostly Ar).

Two discharges are of special interest:

A) The triggered self-sustained magnetron sputtering without a gas burning is the plasma of ionized sputtered atoms [5].

B) The high power pulsed impulse magnetron sputtering HiPIMS burning in a discharge consisting of a mixture of ionized argon with a high ionization of the sputtered atoms [6], [7], [8]. This higher ionization level of the magnetron sputtering process opens up new possibilities to tailor coating properties. The HiPIMS processes have different trade names: HIPIMS, HIPIMS+, HPPMS, MPP, HIPAC and others. HIPAC is used by Sulzer Metaplas: High Ionized Plasma Assisted Coating.

A demand of R&D and production systems to run HIPAC processes is obvious. The solution provided by Sulzer Metaplas is the DOMINO platform (mini, S, L type), see Fig.1. The systems can be configured with both HIPAC and APA arc evaporators as hybrid systems.

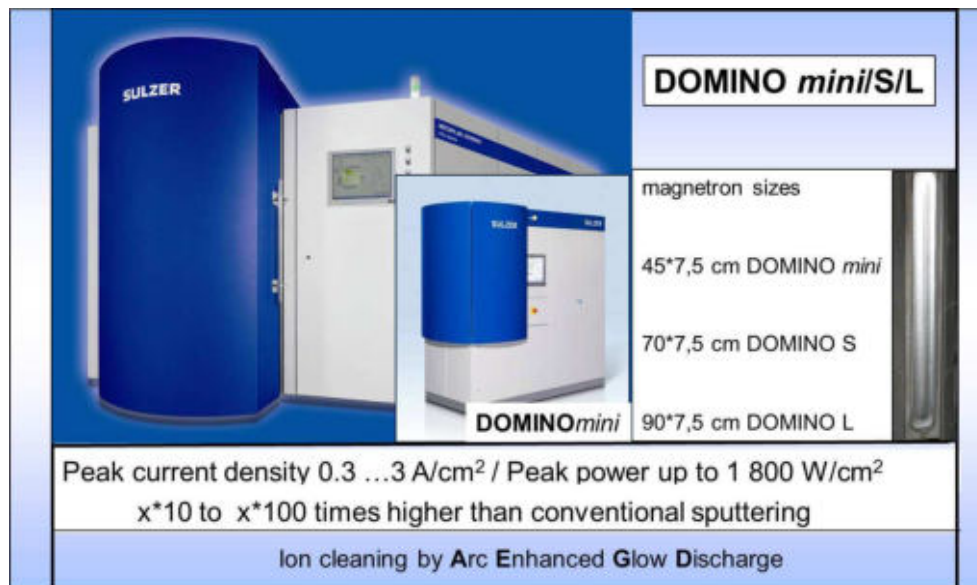


Fig.1: METAPLAS-DOMINO platforms suitable for HIPAC processes, arc processes and for the HI3 hybrid set up with different chamber and magnetron sizes

The HIPAC processes can be processed either in a unipolar discharge mode using one or more magnetrons or in a bipolar discharge mode if at least two magnetrons are installed, see Fig. 2.

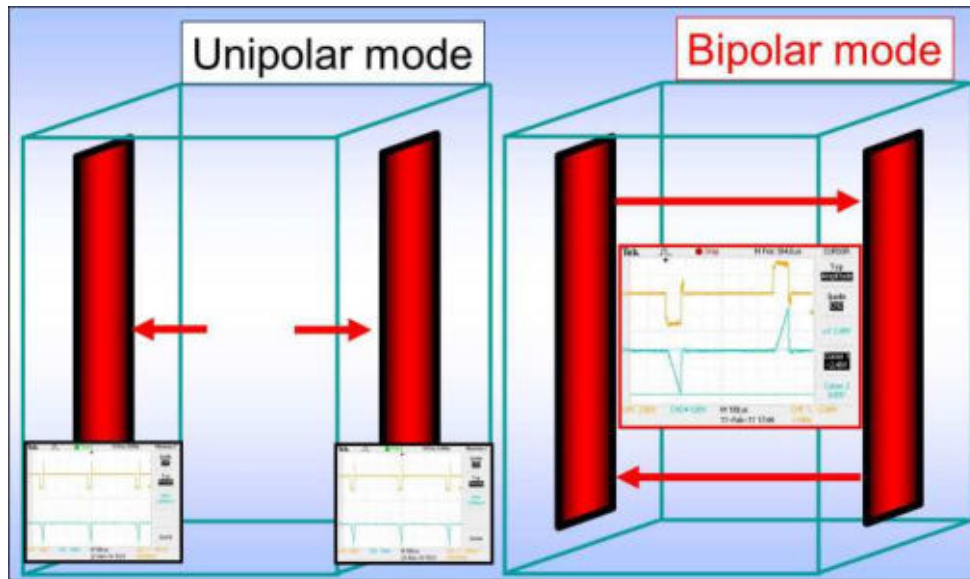


Fig.2: Operation modes of HIPAC within METAPLAS-DOMINO platforms with two magnetrons

Brief description of the HI3 deposition process

The HI3 deposition process combines specific features and advantages of the APA arc and of the HIPAC processes shown in Fig. 3. The ion cleaning is carried out by the powerful AEGD process. The arc evaporation has some limitation to evaporate materials. Mostly metals and its alloys are evaporated. Furthermore, it is not possible to eliminate all droplet emissions in the productive direct arc mode (only almost in the filtered arc mode). The amount of droplets is acceptable for most of the applications (tool, components, decoration). HIPAC has the advantage that it is also possible to atomize and ionize materials which are not possible to evaporate by arc like Si, B<sub>4</sub>C and more, others materials which show a high droplet emission. Thus the HI3 process is suitable to generate new advanced coatings like top layers on arc layers by HIPAC, doping of arc deposited layers within a layer stack by HIPAC, deposition of nanolayers running arc and HIPAC with optimized process parameters at the same time.

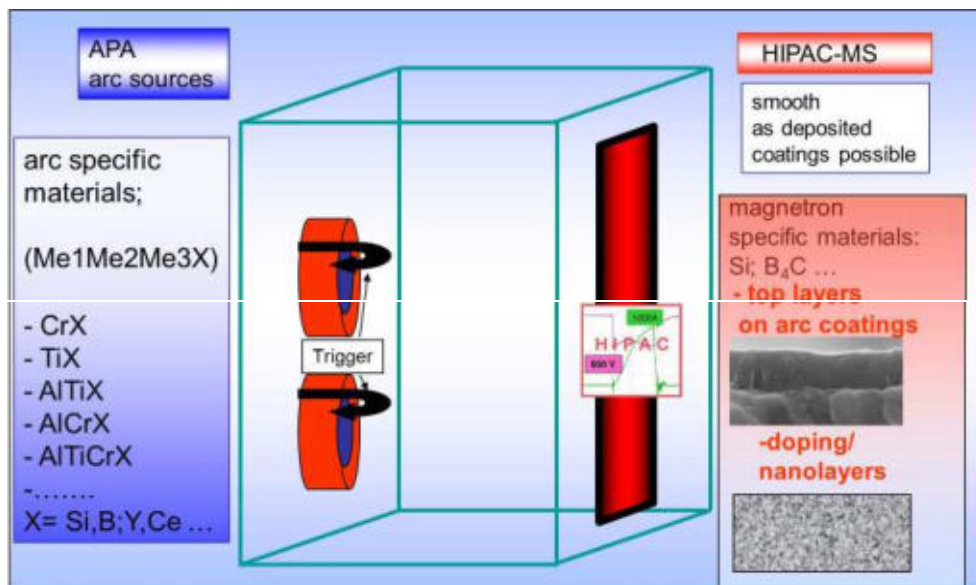


Fig. 3: Combination of specific features and advantages of arc evaporation and HIPAC within the METAPLAS-DOMINO platforms

Preliminary results of the pure HIPAC process in comparison with the pure arc process as well as of the hybrid process, the HI3, will be presented.

## 2. Experimental set up

### 2.1. Comparison Arc and HIPAC

Two important industrial coating types were deposited: AlTiN coatings using a target composition developed by Sulzer Metaplas (Al66at%Ti34at%) [8] and CrN coatings using pure Cr targets. Two different deposition methods were applied: magnetron sputtering and arc evaporation. Magnetron sputtering of the AlTiN coatings was made in the DOMINO mini using two magnetrons mounted in a closed field configuration. The magnetron with an unbalanced magnetic field set up had a size of 7.5 cm in width and 450 mm in length. The used pulse unit allowed a maximum voltage of 1000V/1000A. The magnetron discharge was running in 3 different modes: two magnetrons in the DC mode, one magnetron in DC mode and the other in HIPAC-mode, both magnetrons in the HIPAC-mode. The distance between the substrates and the target surface was 60mm. The bias voltage was set to 50 V, the total pressure was about 1 Pa, Ar:N2 flow of 120:120 sccm. The average power applied to each target was constant with 7 KW (20W/cm<sup>2</sup>). Fig. 4 illustrates the different pulse modes.

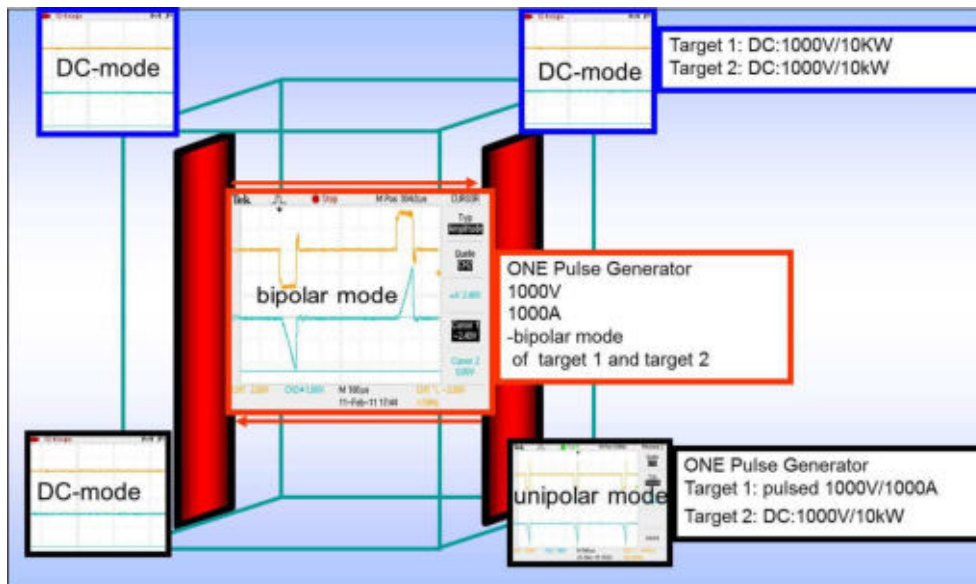


Fig. 4: Possible modes of magnetron sputtering in the DOMINO systems

To compare the growth rate with the growth rate of the arc evaporation also AlTiN coatings were deposited by industrial standard parameter. The distance to the substrate holder was 140 mm. CrN coatings were deposited by HIPAC and vacuum arc to compare the adhesion level and roughness of the coatings in a DOMINO S system. The deposition parameters were the following: total pressure 0,9 Pa, Ar:N2 flow of 250:80 sccm, bias 50V, pulse parameters 150  $\mu$ s on 1500  $\mu$ s off ( f= 606 Hz). The CrN arc coatings were deposited under industrial standard parameters. The ion cleaning was carried out by AEGD (Arc Enhanced Glow Discharge) before HIPAC and arc evaporation [10].

### 2.2. Advanced hybrid coatings: the HI3 process

Two different groups of advanced hybrid coatings were deposited in a DOMINO S to improve the AlTiN base coating deposited by APA arc shown in Fig. 5.

#### 2.2.1. Improve the oxidation stability by a protecting top layer of the type SiBNCO and by doping

It was presented by M. Isaka and J. Vetter, that the top coatings based on SiBNCO deposited by RF sputtering on AlTiN base coatings decreased significantly the oxidation depth ( 950°C/ 3h in air): the oxidation depth of about 60nm was 85 times lower than that of the Al<sub>55</sub>Ti<sub>45</sub>N arc coating and a factor 20 lower than that of the Ti<sub>80</sub>Si<sub>20</sub>N arc coating [11,12]. Recently, SM reported about the development of the HI3 process to deposit such high oxidation stable coating [13,14].

**2.2.2. Improve the self lubrication at high operation temperature by VXN top layers and doping**

It was reported that the formation of vanadium oxide at temperatures of about 600 °C reduces significantly the solid state friction [15]. Moreover we observed a rather high droplet emission of V-droplets in the arc process. The two aspects were the driving force to generate V-containing coatings by the HI3 process.

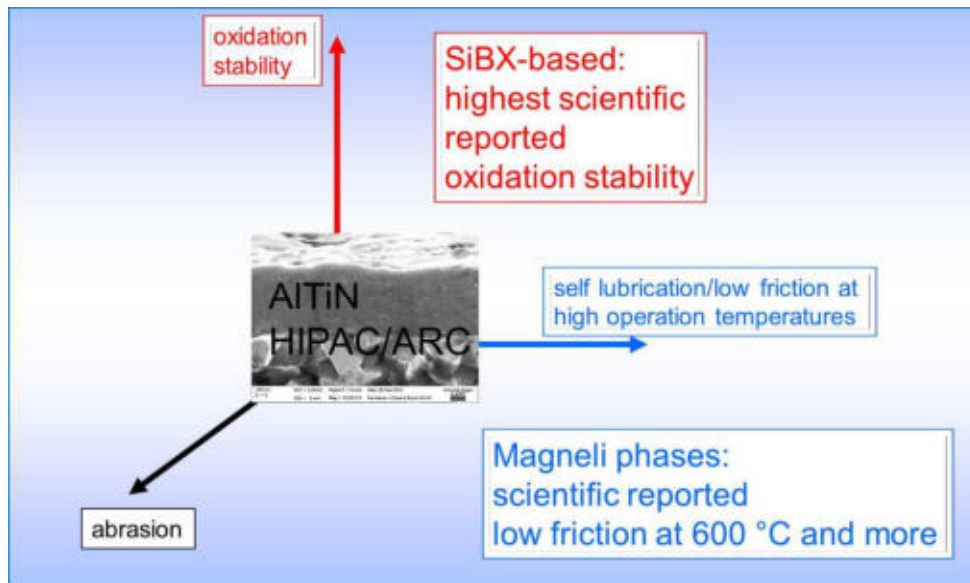


Fig.5: Development direction of advanced hybrid coatings on the base of the HI3 process

**3. Results**

**3.1. Deposition rate of AlTiN coatings in HIPAC-mode**

Fig. 6 shows the deposition rates achieved by magnetron sputtering in the different operation modes. The highest deposition rate was measured for DC-sputtering (14 A, 500V) with a current density of 0.04 A/cm<sup>2</sup>. A decrease was observed when operating in the mixed mode; one target with DC-MS and the other by HIPAC. A sharp drop was measured when a high peak power was applied at both magnetrons running in the HIPAC. The pulse parameters for the high ionized plasma operation were 80 μs on, 500 μs off (f=1724 Hz). The maximum peak current with 600 A results in a current density of 1.8 A/cm<sup>2</sup> and the maximum peak power of about 1 170 W/cm<sup>2</sup>.

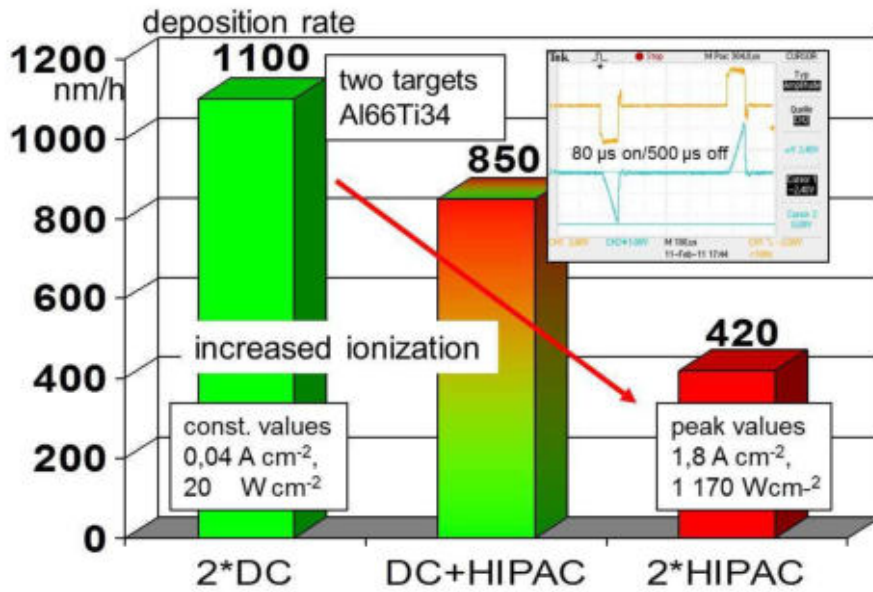


Fig. 6: Deposition rate of AlTiN in different sputtering modes (3-fold rotation)

### 3.2. Energy consumption and deposition rate

A standard industrial (Al66at%Ti34at%)N coating was deposited by two APA evaporators in order to compare the energy consumption to get a specific deposition rate of the different sputtering modes with the energy consumption of the arc evaporation. The same substrate holder was used (3-fold rotation). The deposition rate was measured over a height of about 310 mm (magnetron: ca. 70% of the target length, arc: ca. rim of the arc evaporators). The measured deposition rate [nm/h] was divided by the total energy consumption of the particle sources [KW]. The results are shown in Fig. 7.

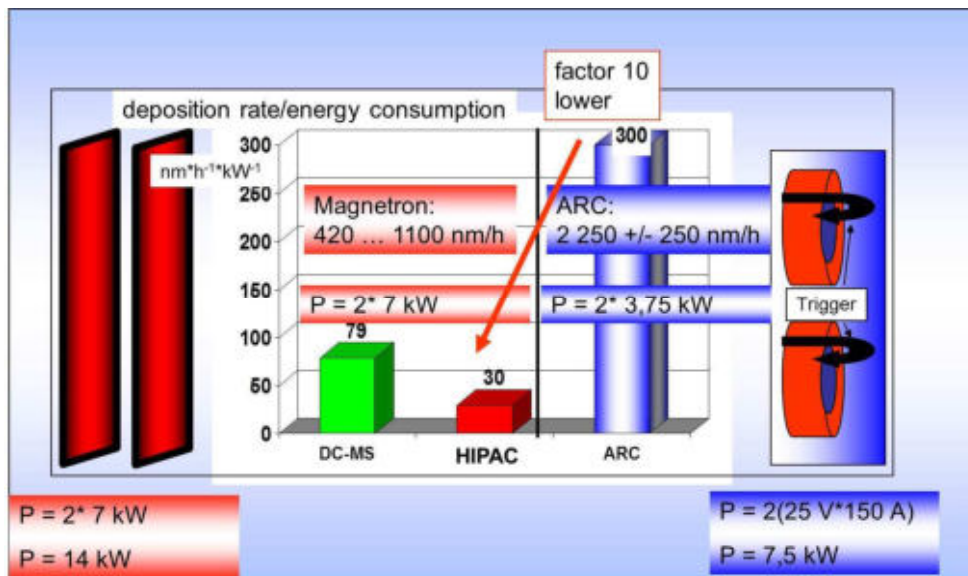


Fig. 7: Deposition rate divided per energy consumption of different sputtering modes and arc evaporation

Obviously the energy consumption to get the same deposition rate increases if the sputtering is switched from the DC- mode to the high ionized HIPAC. The energy consumption to get the same deposition rate increases by a factor of 2.5. The outstanding low energy consumption of the arc is

significant. The required energy to get the same deposition rate like the HIPAC is up to 10 times lower than for the HIPAC.

### 3.3. Roughness

The adhesion and the roughness for CrN coatings deposited by HIPAC and arc evaporation are shown in Fig. 8. Both coating methods show the same high adhesion achieved after AEGD-ion cleaning. The roughness is typically much lower for the HIPAC coating than for the arc coating. The roughness of the arc coating decreases significantly when applying an industrial standard post treatment. However it is still slightly higher than that of the HIPAC coating.

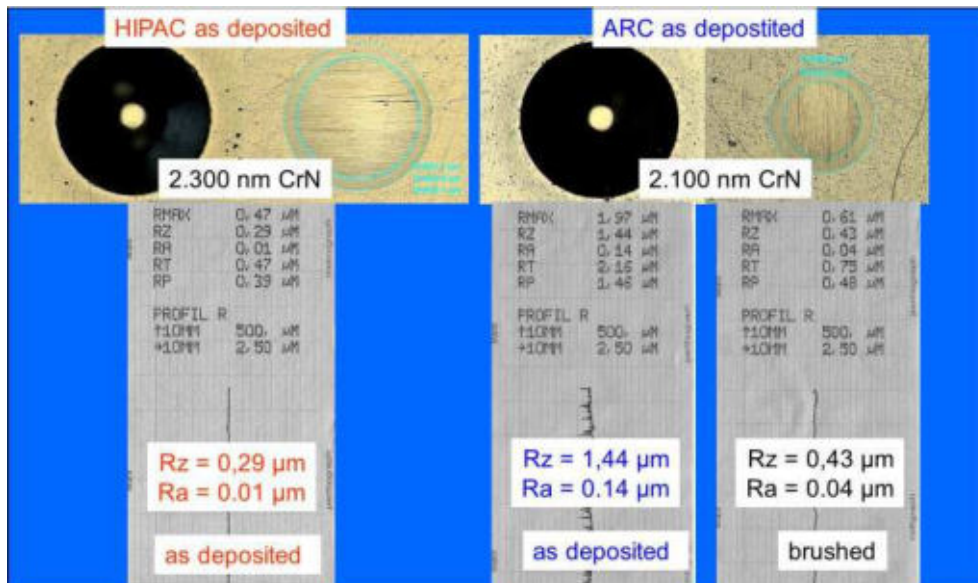


Fig. 8: Roughness of as deposited CrN coating: HCP-MS in as deposited state, arc in as deposited and post treated state

### 3.4. Selected properties of advanced hybrid coatings by the HI3 process

#### 3.4.1. SiBX-based coatings

Fig. 9 shows the coating architecture of a coating with a SiBX top layer. It is obviously that the AlTiN base coating grow with a columnar structure. The hybrid coating deposited by parallel operation of HIPAC and arc shows a grain refinement. The SiBX top layer is amorphous. An unusual structural gradient was generated: from columnar growth step by step to the amorphous growth. The top layer had a nano-hardness of about 2 000, nearly the same hardness as CrN.

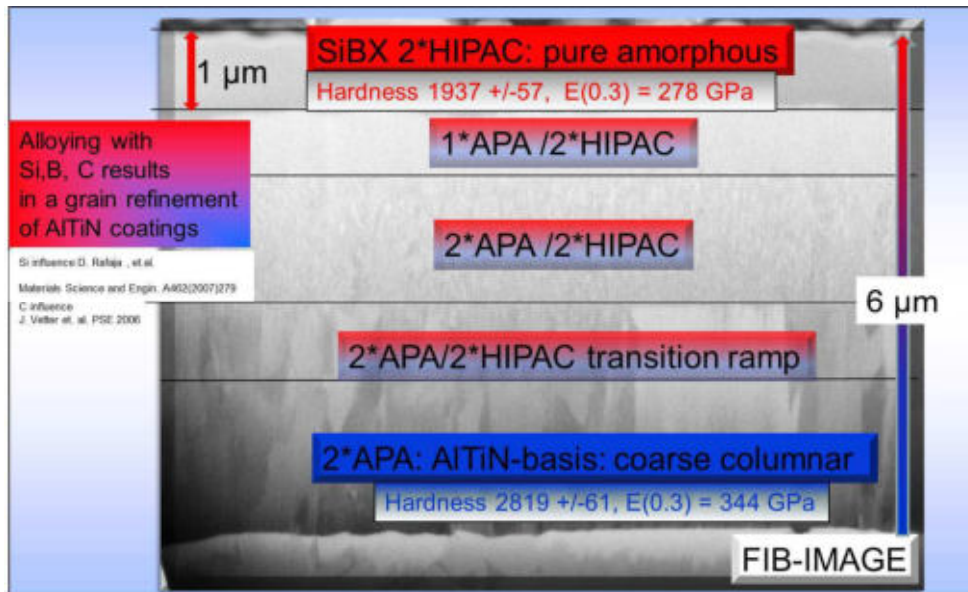


Fig. 9: Morphology and hardness of the coating combining SiBX by HIPAC and AlTiN by arc using the HI3 process

### 3.4.2. VX- based coatings

Fig. 10 shows the coating architecture of a coating with a VXN top layer. It is obviously that the AlTiN base coating grew with a columnar structure. The hybrid coating deposited by parallel operation of HIPAC and arc shows nearly the same columnar structure in contrast to the grain refinement observed for SiBX-base coatings. The VXN top layer grew is also dense columnar, a result for the HIPAC operation. The top VXN layer had a nano-hardness of about 2 300, nearly the same hardness as TiN.

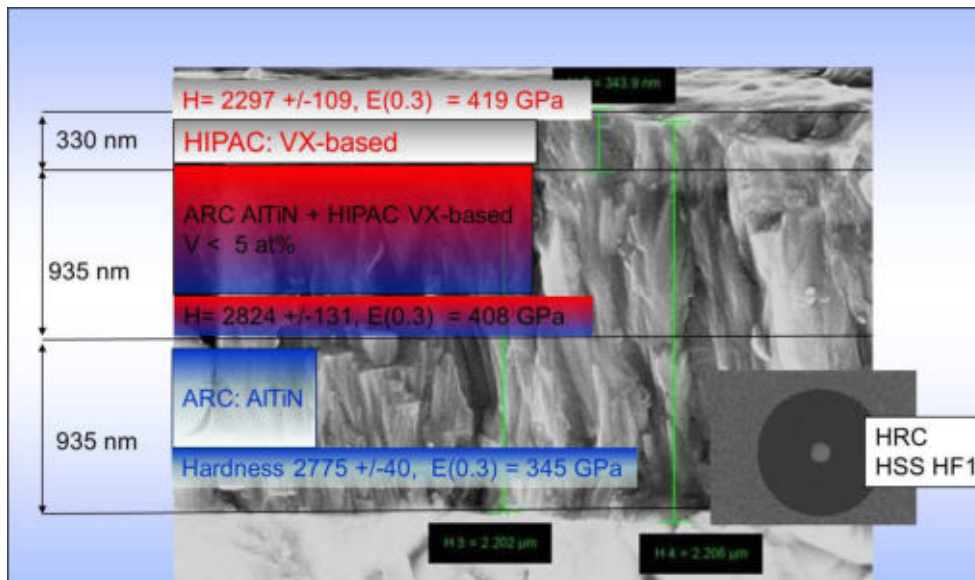


Fig. 10: Morphology and hardness of the coating combining VX by HIPAC and AlTiN by arc using the HI3 process

First tooling application tests were realized. The coating showed surprising good results in cutting of Ti alloys, but also in punching of stainless steel.



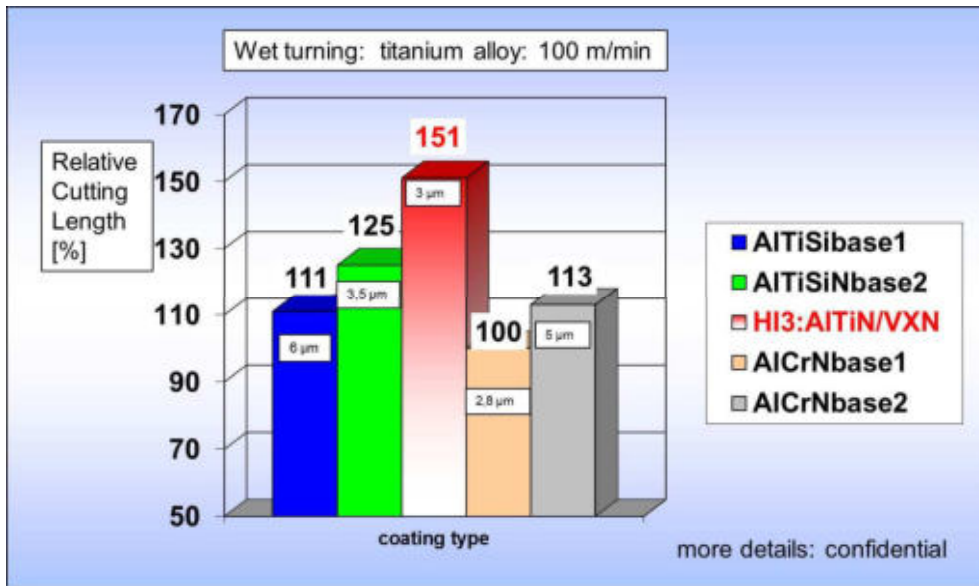


Fig. 11: Cutting test of a coating combining VX by sputtering and AlTiN by arc using the HI3 process

#### 4. Discussion

Table 1 shows selected general process data's and industrial relevant data's for DCMS, HIPAC, arc evaporation and HI3. In addition to the well known data's the energy consumption is added. It has to be pointed out that this comparison is made under the view of industrial applications of hard coatings for wear and friction reduction.

##### 4.1. What are the advantages of HIPAC compared to Arc for wear/friction reduction coatings?

A) It has been shown that HIPAC is able to produce well adherent and smooth coatings. That means for micro tools and components it is an alternative to the filtered vacuum arc evaporation. It should be mentioned, that the low roughness requires both an optimization of process parameters and a clean surrounding inside and outside the chamber, otherwise growth defects will be generated by dust particles [16].

B) The target material variety is larger than for the arc evaporation. Thus HIPAC is the solution for target materials which are difficult or not evaporable by the vacuum arc evaporation, e.g. TiB<sub>2</sub>, SiC, B<sub>4</sub>C.

##### 4.2. What are the disadvantages of HIPAC compared to Arc for wear/friction reduction coatings?

A) It has been shown that the deposition rate is significantly lower than that of the arc evaporation. The shown decrease of the deposition rate of HIPAC in comparison to DCMS is caused by the high power densities applied in the described experiment. The process parameters shall be tailored to apply an optimum of power densities for the required coating properties and by adjusting the reactive gas process to maximize the deposition rates.

B) In addition, the energy consumption to get the same deposition rate is up to a factor 10 higher than for the arc evaporation. This tendency was also shown for Cu coating processes.

##### 4.3. Which potential does hybrid systems have?

The DOMINO platform has both the possibility to deposit coatings either by pure HIPAC and its combination with DC-MS or by arc evaporation to find the optimal coating process for different applications. It should be mentioned, that the droplet content and the resulting roughness of an arc

deposited coating is acceptable for a lot of applications without any post treatment. In several applications like for forming tools and plastic moulds a post treatment has to be applied.

#### 4.4. The HI3 process

One not in detail investigated potential of HIPAC is the hybrid process with vacuum arc evaporation. The possibility to use target materials which are difficult or not evaporable by arc opens the window to deposit dedicated nanocomposites by doping or to generate multilayers (also in the nanoscale) and to adjust functional top layers. First steps were done by Sulzer Metaplas.

Table 1: Basic comparison of arc and magnetron deposition including HIPAC (HiPIMS)

	Target material flexibility	Ionization	Rate	Energy efficiency	Main application wear/friction	* Classical hard coatings	*DLC with PVD process
<b>DC MS</b>	++++	+ max 10%	+++	++	DLC parts decoration (tools)	5%	99 %
<b>HIPAC</b>	+++	++ may exceed 50% [7]	++	+	not defined smooth and dense coatings	?	?
<b>DC Arc</b>	++ limited arc materials	+++ up to 100 % [2,3]	++++	++++	tools, components (decoration)	95%	1%
<b>Arc Filter</b>	++ mostly carbon	++++ 100%	+	+	Components (tools)	less 1%	less 1%
<b>HI3 = HIPAC &amp; Arc</b>	+++	++ & +++	++ & ++++	+ & ++++	tools, components	Advanced Coatings	Advanced Coatings

\*percentage of application is estimated from market observation, not based on statistical evaluation

#### 5. Summary and conclusions

1. The DOMINO platform gives the possibility to investigate specific process parameters and coating properties for DC-MS, HIPAC-MS in comparison to the arc evaporation.
2. The DOMINO platform allows to run HIPAC-MS and vacuum arc evaporation as a hybrid process.
3. The deposition rate of HIPAC-MS is lower than that of DC-MS and significantly lower than that of the vacuum arc in the investigated configuration.
4. The energy consumption to get a specific deposition rate is for HIPAC-MS higher than for DC-MS and much higher than for the vacuum arc evaporation (up to factor 10).
5. Two advanced coating systems were successfully realized by the HI3 process: a coating architecture based on Si<sub>3</sub>N<sub>4</sub> by HIPAC to increase the oxidation stability and , a coating type based on TiN by HIPAC to decrease the high temperature friction.

#### References

- [1] Vetter, J., Müller, J. Erkens, G.:Domino Platform: PVD coaters for Arc Evaporation and High Current Pulsed Magnetron Sputtering, IOP Conf. Series: Materials and Engineering 39(2012) 012004
- [2] Siemroth P., Schultrich B., Schülke T., Surf. Coat. Techn. 74-75 (1995)92-96
- [3] Rother B., Vetter J., Wiss. Z. d.TU Karl-Marx-Stadt 31(1989) H3, p. 433-445
- [4] Boxman R.L., Martin P.L., Sanders D.M.: Handbook of vacuum arc science and technology, Noyes Publications, New Jersey, 1995
- [5] Dorodnov A.M, Petrosov B.A, Sov. Phys. Tech. Phys 26(3)1981, 304

- [6] Khodachenko G.V., Mozgrin D.V., Fetisov I.K. patent RU2058429, 1993
- [7] Helmersson U., Lattemann M., Bohlmark J., Ehiasarian A.P., Gudmundsson J. P., Thin Solid Films, 513( 2006) 1-24
- [8] Anders A., Surf. Coat. Techn. 204(2010) 2864-2868
- [9] Vetter, J., Journal of Advanced Materials. Band 31, Nr. 2, 1999, S. 41–47
- [10] Vetter J., Perry A.J., Surf. Coat. Techn. 61(1993)305-309
- [11] SiBNCO coatings for High Performance Applications, presentation conference ICMCTF, USA, San Diego, 2010
- [12] A new class of coatings for cutting tools: non oxide ceramic coatings, presentation: conference PSE, Garmay, Garmisch-Partenkirchen, 2010
- [13] Vetter, J. , Müller, J., Erkens, G.: Hybrid – PVD coatings: ARC evaporation combined with HIPAC, presentation conference ICMCTF , USA, San Diego, 2012
- [14] Vetter, J., Müller, J., Erkens, G.: Advanced PVD coatings: arc evaporation combined with HIPAC presentation: conference PSE, Germany, Garmisch-Partenkirchen, 2012
- [15] Gassner, G., Mayrhofer, P.H., Kutschej, K. , Mitterer, C., Kathrein, M. A New Low Friction Concept for High Temperatures: Lubricious Oxide Formation on Sputtered VN Coatings, TRIBOLOGY LETTERS, Volume 17, Number 4 (2004), 751-756
- [16] Vetter J., Stüber M., Ulrich S., Surf. Coat. Techn. 168(2003) 169-178