High Rate Oxide Deposition onto Web by Reactive Sputtering from Rotatable Magnetrons

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There is a strong market demand for high rate deposition of oxides onto flexible substrates. The primary sectors for such material span established and new growing markets:

1. Touch panel film – reactive SiO_2 layers for anti-reflection
2. TCO layers for flexible solar cells – aluminum doped zinc oxide
3. High gas barrier film for PV and OLED applications – aluminum oxide

These layers are predominately deposited by magnetron sputtering. In the case of SiO_2 and Al_20_3 the process is a reaction between the metallic target material and oxygen gas. Currently for the TCO layers in solar cells, generally the deposition is the non-reactive from a ceramic target material with a composition similar to the chemistry of the final layer.
This presentation focuses on reactive deposition methods and the means to increase rates via process feedback control. In addition, the advantages of rotatable magnetrons as opposed to planar magnetrons will be presented. This will focus on the inherent benefits but also means to reduce the heat load on the flexible web by the means of magnetic confinement of electrons via an active anode or magnetic coupling of two cathodes.
Examples of process and film data for these three important market sectors will be presented.

Introduction

Magnetron sputtering is the most widely used technique for creating a flux of vapour for the vacuum deposition industries. The flux of vapour is generated by a magnetically confined plasma over the 'target' surface which 'sputters' the target material ('target' is the name given to the source of the material). The common geometry of source material is a flat plate like target. In recent years a new form of magnetron source has become much more popular. This type of magnetron is known as a rotatable magnetron and uses a tube type target as the source of the vapour – see figure 01.

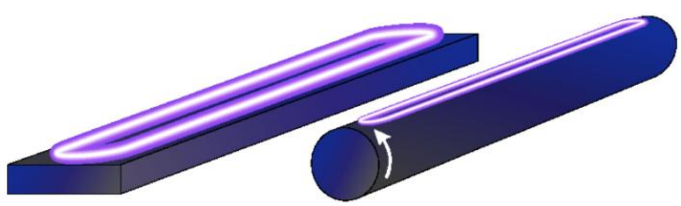


Figure 1. A standard planar target compared to a rotatable target geometry.

The rotatable magnetron has advantages of better material usage and longer continuous operation. The use of rotatable magnetrons has increased recently as the solar and display industries look for greater efficiencies and cost reduction.

Reactive Sputtering

The concept of reacting a sputtered species with a reactive gas is a well established technique for the creation of compound films in the architectural glass and display markets. For example, all touch panel flexible film is created in such a mode with the silicon being sputtered in the presence of oxygen. There is a large amount of literature covering the reactive sputtering concept, hence this paper will deal with the control of such processes. Feedback control of the reactive gas is a means to increase rates of what is an unstable process when operated in the high rate ‘transition’ mode. Recent advances in the ability to control processes with great speed and accuracy has greatly increased the competitiveness of this mode of film production.

Process Control

Most industrial plasma processes such as coated or treated web-based products are dependant upon some form of control of plasma properties for repeatable and reliable production. Process control involves all the aspects of the vacuum equipment, substrate preparation, plasma source condition, power supplies, valves (inputs / outputs), signal and data processing and the user's understanding and ability.

Understanding the limitations of each of the parameters affecting the process is the first step towards control. Without quantification of the limitations of a system, attempting control could be a frustrating exercise. This is true for even the simplest of products but becomes critical where more advanced flexible thin film products are concerned.

In some cases, the manufacturing of marketable coating structures require an accurate control of the various parameters in a reactive gas environment. Commonly in these circumstances the plasma is not stable if all the inputs and outputs of the system were to remain constant. The ideal situation is to move a process from set-point A to B in zero time and maintain the monitored signal with a fluctuation equal to zero. For large scale production with plasma zones of several meters in length this ideal is not achievable, but fast stable control can be achieved.

Gencoa has been active in reactive sputtering control for 15 years and this paper will illustrate the degree to which these advances have improved production of compound layers. This has been achieved by the continuous development of a closed-loop control system and appropriate sensors which feedback the relevant information necessary to control the process.

It is now possible to automatically control the critical parameters in the process to prevent significant variations. The system uses a wide range of sensing capabilities to monitor and pro-actively manipulate the process parameters to maintain stability of the whole environment and maximize production speeds. The tuning of such as process controllers typically requires some element of knowledge and skill. This is an area that has lead to a reluctance on the part of the operators to adopt this technology. However, sophisticated software’s features (see figure 2) and introduction of an auto-tuning system has largely removed these barriers for the up-take of the technology.

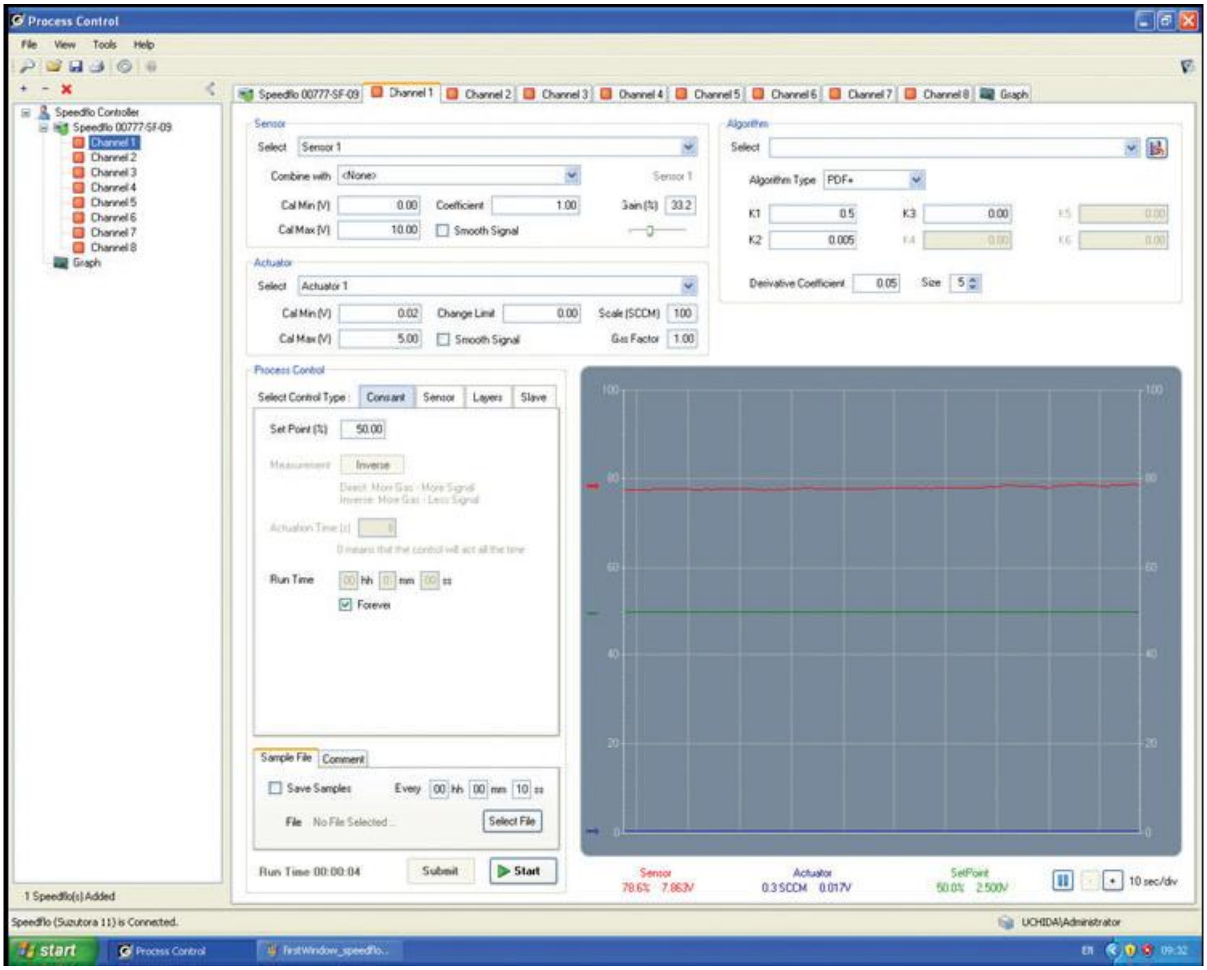


Figure 2. Visualization of the features of the process control window for the Gencoa Speedflo reactive gas feedback controller.

The primary reason to adopt a feedback control system for reactive gas input is to enable the target to run in a metal mode whilst introducing just the right amount of gas to enable the correct coating stoichiometry to be achieved. Typically in vacuum coating, the gases are input into processes with great accuracy by means of mass flow valves (MFC's) that give a constant flow of gas into the sputtered coating flux. The reactive sputtering process is however highly unstable and the target surface displays rapid changes from the pure metal mode into a 'poisoned' target surface state. When a sputter target is poisoned, the surface is covered in the reaction product of the gas and the metal. The results is that the rate of sputtering drops greatly due to the low sputter yields of the reacted metal layer in comparison to the pure metal layer.

For stable operation when performing reactive sputtering there are 3 options:

1. Run the process in a full poisoned low rate state all the time by introducing excess reactive gas.
2. Pump the vacuum chamber at very high speed that allows MFC control alone to achieve some stability in the transition mode. This is expensive or unrealistic in large process chambers.

3. Use a process controller that automatically adjusts the amount of reactive gas at to ensure that the target surface is held in a metallic mode to deliver constant high rates.

The cost benefits of the third controlled option approach far outreaches what is commercially possible with either of the other two approaches. This is illustrated in the following example.

Right is an example of a schematic of a reactive sputtering module from the architectural glass industry. The sputtering cathode is a dual rotatable with a 1.55m target length with an applied power of 25kW MF power (AC mode – switching power +ve and –ve between each of the 2 targets). There are 3 plasma emission monitoring (P.E.M.) based sensors positioned down the target length to monitor the level of oxygen gas in the process zone. The oxygen gas is delivered to gas bars that combine high speeds of response and the ability to adjust the uniformity over the glass width to <±1.5%.

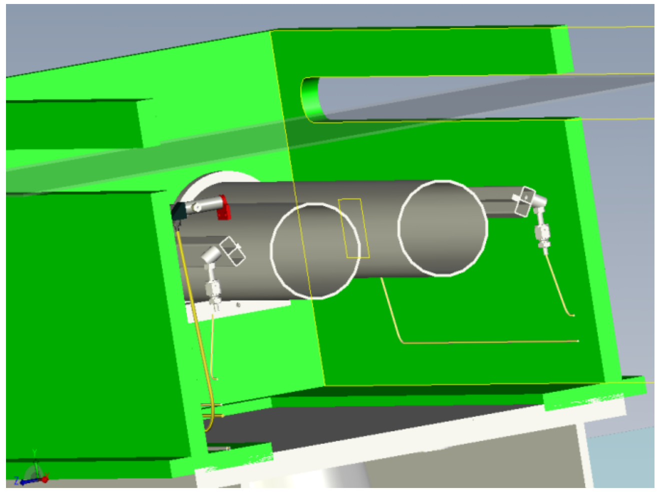


Figure 3. Schematic layout of sputter-up dual rotatable silicon cathodes with P.E.M. monitoring and gas delivery bars.

The deposition rate of SiOx layer is displayed in figure 4. On the vertical axis is the thickness of material deposited in nm for a piece of glass travelling at 1m/min past the dual cathodes. The horizontal axis is the setting of the oxygen level as measured by the plasma emission of the oxygen gas at 777nm wavelength of light - 100% is an oxygen signal representing fully poisoned mode of excess oxygen, and 0% is the pure metallic state with no oxygen present in the plasma signal.

The process controller can achieve stable operation anywhere from metallic to poisoned modes. The rate varies from 20nm.m/min in the poisoned state to 100nm.m/min in the near metallic state. Typically, fully transparent films can be found in the range 40-65nm.m/min. Films deposited at higher rates will have some levels of absorption and may not be useful for all products.

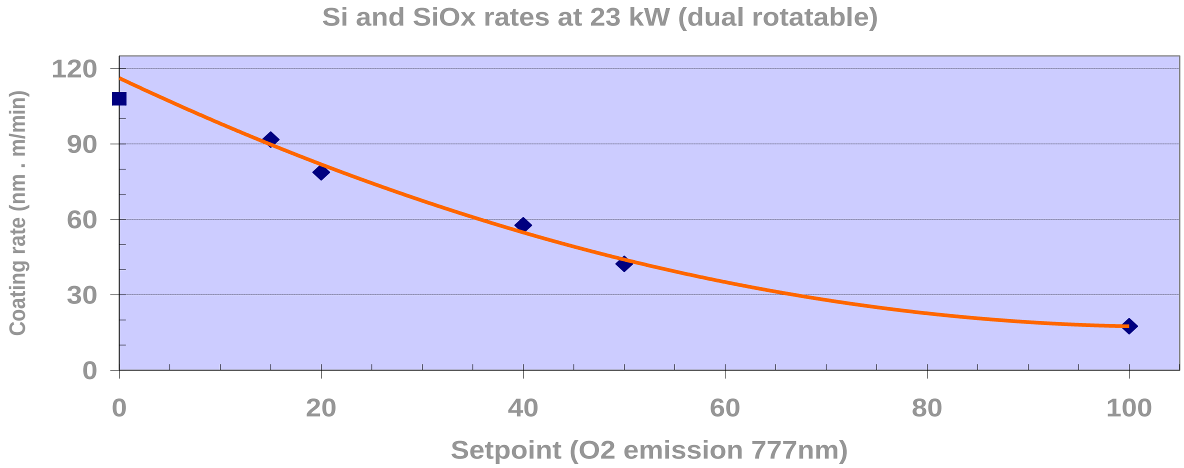


Figure 4, Variation of dynamic deposition rate for SiOx layers with respect to P.E.M setpoint

This method for rate enhancement is available for all metal and oxygen systems and offers productivity improvements of 3-4 times when compared to systems run in 'open-loop' uncontrolled mode. Other layers types that commonly benefit from 'closed-loop' feedback control in industry are Al_2O_3, Nb_2O_5, TiO_2, ZnO.

Uniformity adjustment

Along side the need for high rates comes the desire for good film uniformity. When a target is run in fully poisoned mode, the distribution of reactive gas is not so critical as there is an excess in the chamber and all of the target surface is in the same compound state.
For reactive sputtering in the transition mode however, the balance between the metal and gas needs to be same across the substrate area to have the same thickness of layer, stoichiometry and layer properties. A uniform distribution of gas in a pipe can be achieved by a large cross-sectional area allowing a fast lateral spread and minimal pressure drops down the distribution tube. The outlets need to deliver the same gas input to the system from each point, hence need the same and precise conduction from each oulet.
The above requirements for a uniform flow has to be balanced with the need for 'speed' of gas delivery. As the system relies upon a feedback from the process, a reduction of the time delay will lead to better levels of control. Typically a closed loop response time of 10 msec or less is required to control fast response material systems such as SiO_2 and Al_2O_3. For the combined Gencoa Speedflo gas controller and gas bar delivery system with plasma emission monitoring as the sensor, the following are the typical response times of the different elements:

1. Signal detection from the plasma to the photo-multiplier – speed of light.
2. Internal processing of the signal in the Speedflo unit and delivery of the signal to the mass flow valve, <1 msec.

3. Response of the MFC to the set-point change – 2 msec
4. Delivery of the gas from the MFC outlet to the area in-front of the cathode – 3 to 10 msec depending upon system size.

It is always found that the gas lines and the gas bar represent the largest delay in the system. Consequently, this part of the system is very important as the balance of uniformity and speed is paramount. Figure 4 illustrates such an optimized gas delivery arrangement with a fast response and the ability to 'trim' the gas contribution over 3 different zones down the length of the system.

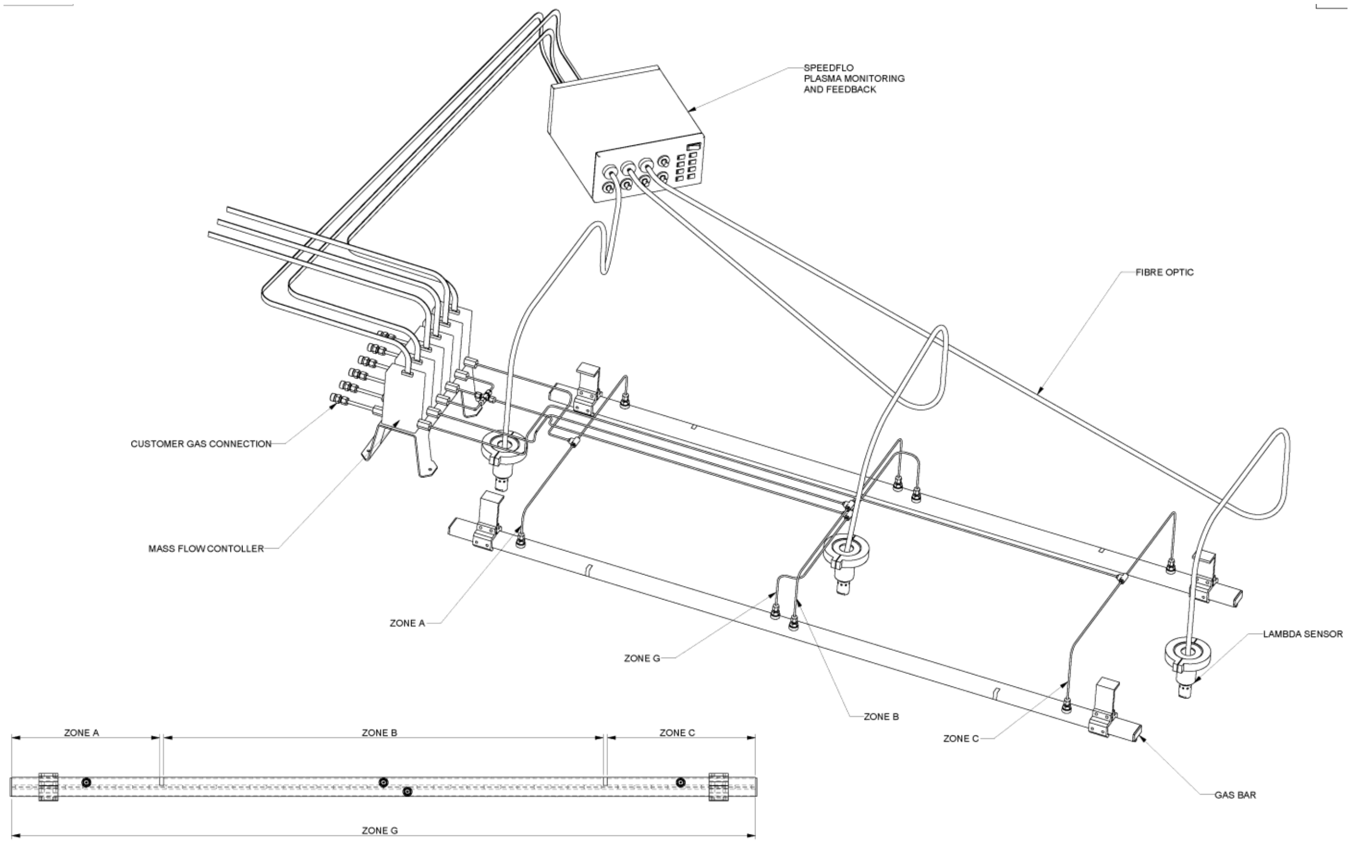


Figure 5. A typical gas delivery system capable of a <10 msec response time and film uniformity adjustment.

The uniformity required for each application will vary depending upon the requirements of the layers. Typically the range is ± 1 to 5% with the best uniformity required for double and triple low-emission glazing which is required in the 1% range. Over a large area of 1-3 m this is major challenge and requires a good understanding of all elements of the process environment to achieve the required specification. Figure 6 illustrates the adjustment by means of the gas system of the layer thickness from ±2.5% to ±1.5%.

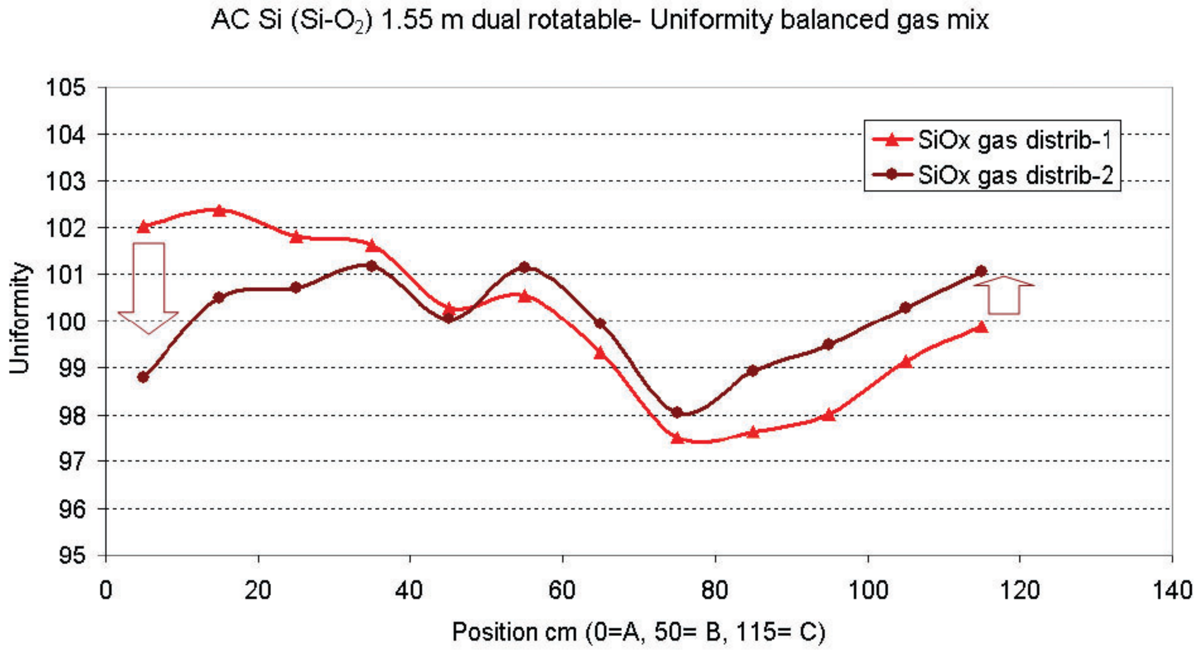


Figure 6. Uniformity tuning over a 1.2m wide glass via adjustment of gas 'trimming' zones.

Medium Frequency Double Cathodes

The final challenge of a reactive sputtering environment is the stability of the power applied to the target. The power needs to remain constant in order to ensure uniform film properties on the product as the substrate will maintain a constant speed past the targets. The oxide layers of interest from a commercial point of view then to be insulators or semi-conductors. As such, when reactive sputtering in the transition mode, the target surface will contain conducting and non-conducting or poorly conducting areas. To avoid electrical breakdown (arcing) between the two areas, the power supply will need to have the ability to eliminate the charge build-up on the non-conducting areas of the target surface. This is achieved by the method of reversing the polarity of the normally negatively charged target surface at a frequency that ensures enough electron current is attracted to the target surface in order to neutralize the positive charge build-up on the dielectric areas present. This pulsing of the voltage into the ground or positive state is widely used to reduce the chance of arcs during sputtering or other plasma processes.

Another potential problem exists when dielectric layers are being created by reactive sputtering. In the same way that some areas of the target surface area are covered with dielectric, all the other elements in the vacuum chamber such as the magnetron dark-space and shields are subject to dielectric layer coverage. As these parts become coated and hence electrically insulating, they loose their ability to provide a stable earth return for the plasma electrons. The plasma is effectively an electric circuit between a cathode (target) and anode (dark-space shield and chamber walls). In order to maintain equilibrium, the electrons present in the plasma are required to be conducted to an anode

in an equivalent number to match the ion current conducted through the negatively biased target. This completes the electrical circuit and ensure stability.
When the system anode becomes coated with non-conducting oxide layers, the ability for the electrons to ‘find’ an anode is affected and instability is quickly observed in the sputtering plasma. This instability leads to poor film quality, arcing and stray discharges. This instability will render a reactive process uncontrollable and in the case of Al_2O_3 the onset of this regime can occur in less than 30 minutes.
There are methods available to ‘hide’ the anode from the dielectric coating flux and guide the electrons to that point. But the most effective and most widely used means available is to adopt the so called ‘double’ magnetron method where the target surfaces alternative polarities and the electron current is drawn in turn from one target to the next, see figure 7.

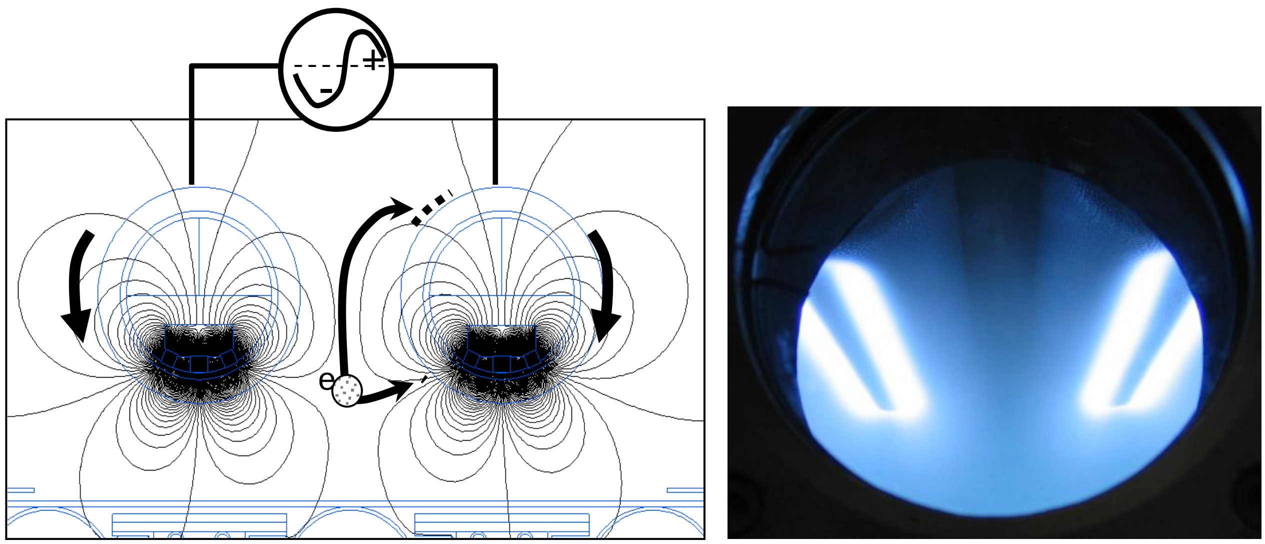


Figure 7. Schematic representation of a medium frequency discharge between two rotatable magnetrons and the resulting plasma (this method shown in figure 7 relies upon IPR covered by a current US patent owned by Von Ardenne Anlagentecknik, FRG).

The technique of two targets working as a pair with AC power between the two for the reactive deposition of dielectric coatings is a critical aspect to the technology and forms a large part of the success of the technique. The principle advantage for reactive sputtering using rotatable targets is the self-cleaning effect as the target tube rotates. This prevents areas of re-deposit building up which is a common problem with planar magnetron as no all areas of the target surface can be sputtered
There is however one drawback with using this method to coat heat sensitive materials and lies in the heat and plasma energy generated when the electrons move from one target to the other. The electron movement at typically 40 kHz between the targets ionizes the argon support gas present in the discharge. Large numbers of ions and electrons will be present in the area of the substrate. In the case of glass substrates this can be beneficial in terms of enhancing the layer properties as a result of the extra energy available for the growth process. However, when coating onto temperature sensitive films, this new source of heat is unwanted and will reduce the possible coating rate as the

energy of condensation in the system needs to be reduced in order to compensate from the plasma heating load. It is hence desirable to reduce this heat load as much as possible and a solution exists.
As electrons are high sensitive to magnetic fields, a method has been developed (patent pending) that uses magnetic guiding of the electrons between the two targets in order to limit the impact of the plasma on the web. Figure 8 below shows such a magnetic confinement in-front of the targets and the effect on the plasma for a reactive aluminium oxide process. The magnetic effect is created by linking the magnetic fields of the 2 cathodes in such a way that the electrons are channeled more effectively between the two cathodes. The advantage of this design are fourfold:

1. Reduced heat load on the web.
2. Less plasma bombardment of the growing film – lowers the resistivity of transparent conducting oxide layers.
3. A more energy efficient lower impedance plasma's resulting in lower energy use.
4. Better coating uniformity – reduced electrostatic charge on a floating substrate.

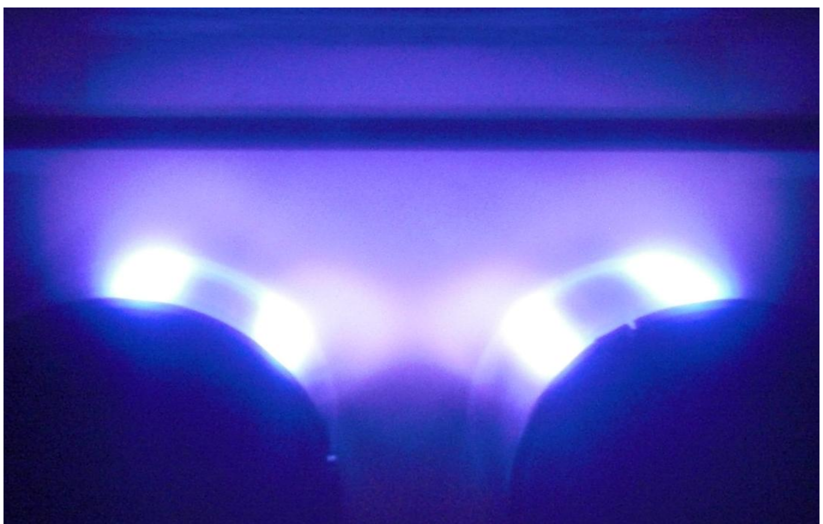
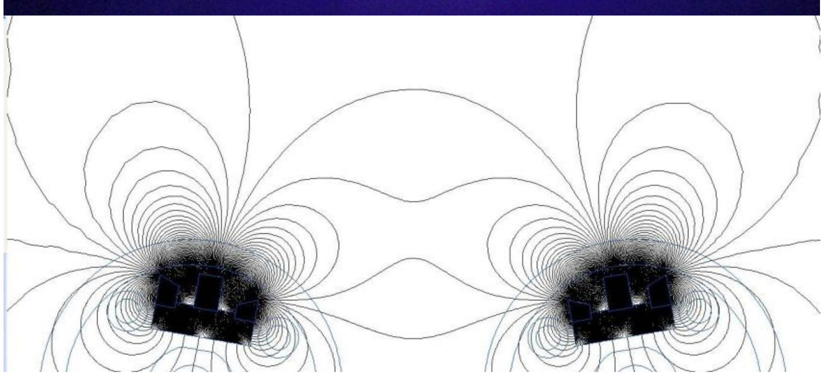


Figure 8.

Plasma distribution in front of a dual rotatable magnetron pair with AC power, aluminium targets and oxygen gas.



Assymetric and linked magnetic field distribution for dual cathode low impedance AC plasma.

The above magnetic design is now being used to very good effect in the creation of a range of different reactive oxide layers from transparent conducting oxides (TCO) to the widely used SiO_2 and more recent Al_2O_3 gas barrier material systems.

Conclusions

A number of strong growth markets in the solar and display sectors demand the use of high rate processes for oxide deposition on flexible substrates. Rate enhancements available from the reactive sputtering method is an ideal means to meet the commercial need for highly uniform and lower cost production methods. The range of sensor combinations available and sophistication of the Gencoa Speedflo control software enables users to achieve optimum conditions with relative simplicity.
Rotatable magnetrons likewise offer advantages for the production of such oxide layers in terms of defect reduction in the layers and a long target lifetime. The inherent problem of increased heat loads from rotatable magnetrons can be counteracted by improved magnetic confinement of the electrons that are present in the plasma. By channeling the electrons away from the substrate, the extra heating can be avoided and high rates can be maintained on temperature sensitive web materials. This method magnetic confinement method also offers improvements in the layer properties such as uniformity and reduced resistivity – in the case of TCO layers.