Sputtering onions

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Dedicated to Roswita, Kobelien, Hannelinde and Griet*

* These four wonderful ladies daily support me but I should thank Hannelinde as editor. The placement of comma's is still not clear to me.

Sputtering onions? This seems to be some kind of strange nerdy question raised by someone too long in the field of sputter deposition. When mounting a piece of an onion into a vacuum system, the part will loose water and dessicate. I never performed the experiment myself, but when checking the literature I noticed others did [1]. I learned that interactions of ions with organic (bio)material are being investigated and have relevance, as in outer space high energetic species can bombard astronauts [2].

However, the main idea behind this curious title is related to research on reactive magnetron sputter deposition [3]. This research is complex and we unravel layer-by-layer the fundamental driving mechanisms, occuring during thin film deposition with this wonderful technique. It is not a coincidence that I mention "layer-by-layer" here as we in some cases grow thin films in this fashion. There is another link to onions... A large part of this book is on ions and the interaction of ions with the cathode of the magnetron discharge, known as the target.

The content of this book is based on a series of LinkedIn posts where each week I presented a playing card introducing one of the figures in this book, and a short explanation. I offer the deck of playing cards as a present to many colleagues to acknowledge our mutual interest in the research field and our stimulating discussions. I received many positive comments on these posts with the request to publish the text and figures. And so, this book is the result.

Diederik Depla
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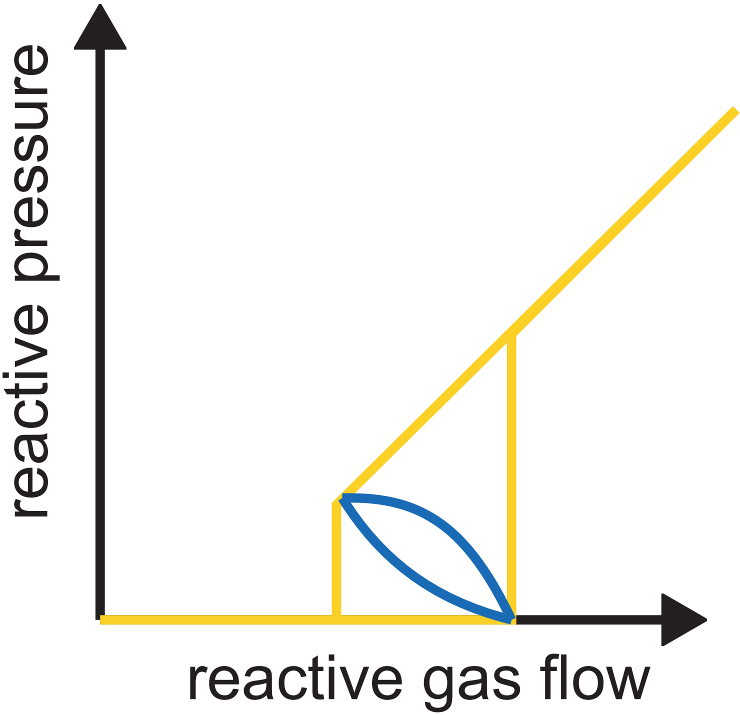
[1] Sangyuenyongpipat, S. et al. Nuclear Instruments and Methods in Physics Research B 227 (2005) 289-298
[2] Feng H. et al. Materials Science and Engineering R 54 (2006) 49-120 [3] The colors refer to the pages dedicated to one of these keywords.

The hysteresis behavior of the reactive gas process parameters, when using flow control, is a typical feature of reactive magnetron sputtering. This is shown in the schematic as the yellow curve. The region inside the loop, known as the transition region, is not accessible under these conditions. This can be circumvented when the reactive gas flow is controlled by a feedback loop, using one (or more) process parameter as input [1]. Under some conditions, it is observed that the process follows a different path from low to high reactive flow than when the experiment is performed in the reverse way. This feature of reactive magnetron sputtering has been dubbed "double hysteresis" [2]. The branch when going from low to high oxygen flow is know as the metallic branch, the reverse as the poisoned branch. The origin of the two paths is related to reactive ion implantation, which is defined by the reactive gas fraction in the discharge. A slight increase of the fraction will result in more compound in the target and a lower target erosion speed. Firstly, this lower erosion speed causes the initial concentration of implanted reactive gas atoms to increase, as they are transported more slowly toward the surface. Secondly, the implanted reactive gas atoms will reside longer in the target due to this slower transport. Both effects enhance the subsurface compound formation, since there is more reagent and more time to react. This, finally, enriches the compound concentration at the target surface, which is an amplification of the original effect. A similar but opposite loop can be described but at a slightly different gas fraction [3].

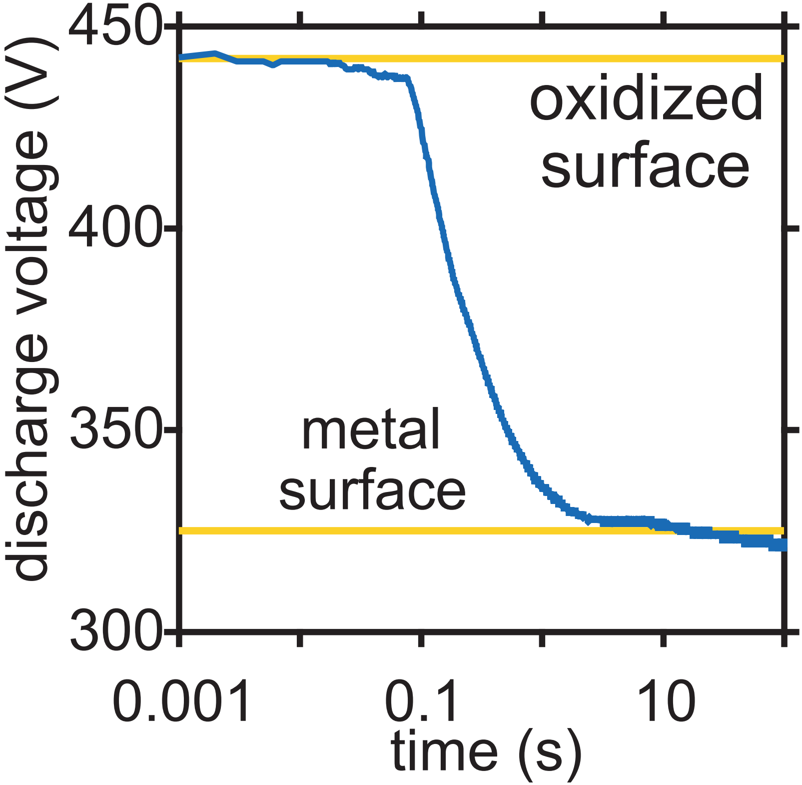
[1] Sproul, W. D. et al. Thin Solid Films 491 (2005) 1-17 [2] Schelfhout, R. et al. Applied Physics Letters 109 (2016) 111605 [3] Van Bever, J. et al. Journal of Physics D: Applied Physics 55 (2022) 355302



double hysteresis

Knowledge of the thickness of the compound layer formed on the target during reactive magnetron sputtering is important to understand the underlying formation mechanisms. Several approaches can be used to access the thickness. The areal density of nitrogen was measured during in-situ nuclear resonance analysis [1]. A silicon wafer was used as a target during RF reactive sputtering, and the formed oxide/nitride layer within the erosion zone was measured by ellipsometry [2]. The target nitridation during reactive magnetron sputtering was studied by ex-situ XPS [3, 4]. Another way to analyze the film thickness is by recording the discharge voltage during sputter cleaning of the target as shown in the picture [5]. A similar thickness, in the order of a few nanometer, was found in all studies. Although still very thin, this thickness elucidates that the layer cannot be formed by only chemisorption, even when assuming a high target temperature [6]. The film thickness corresponds to the implantation range of reactive gas ions, showing that the compound layer is finally formed by reactive ion implantation into the target.

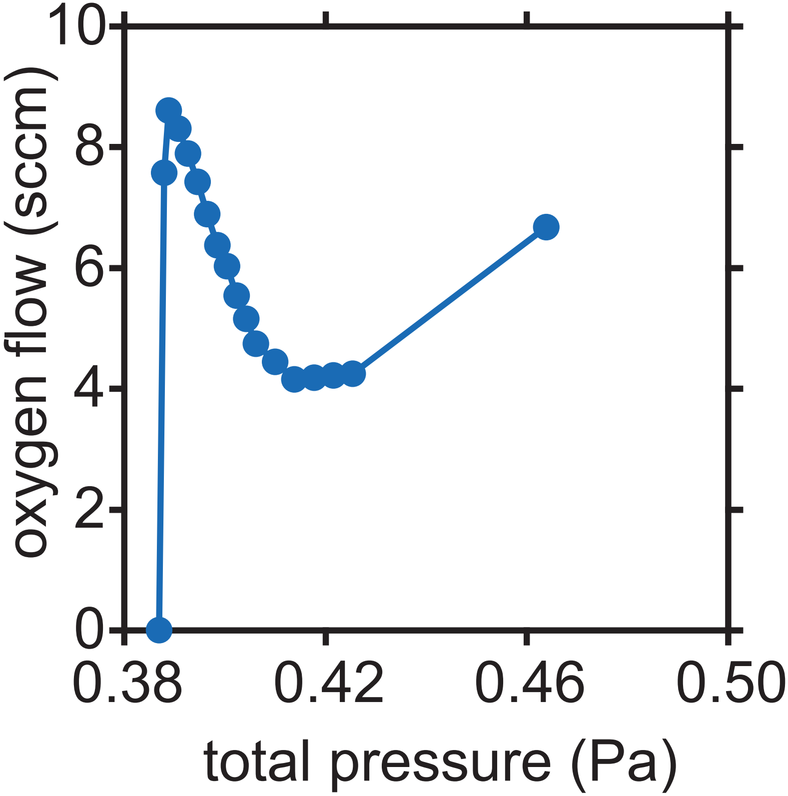
[1] Guttler, D. et al. Applied Physics Letters 85 (2004) 6134-6136 [2] Abe, Y. et al. Japanese Journal of Applied Physics Part 1-Regular Papers Brief Communications \& Review Papers 46 (2007) 6778-6781 [3] Greczynski, G. et al. Applied Physics Letters 111 (2017) 021604 [4] Greczynski, G. et al. Journal of Physics D: Applied Physics 51 (2018) 05LT01 [5] Depla, D. et al. Thin Solid Films 515 (2006) 468--471 [6] Depla, D. et al. Vacuum 217 (2023) 112391



compound layer

The measurement of a process curve during reactive sputtering, showing, for example, the reactive gas pressure as a function of the reactive gas flow, is known as a hysteresis experiment. The measured process curve can show an abrupt change between process conditions known as the metallic and poisoned regime. The deposition of fully oxidized compound layers can only be achieved in poisoned mode, which is hampered by a low deposition rate. This problem can be mitigated by controlling the reactive gas flow through a feedback control loop using another deposition parameter as input parameter. The plot shows an example where the flow is controlled by the reactive gas pressure. This approach allows the deposition of thin films in the transition region which corresponds to the region in the plot with a negative slope. The data of this plot was provided by Bill Sproul, the founder of this approach, who passed away on December 19th 2024 [1]. This page is dedicated to him.

[1] Sproul, W. D. et al. Thin Solid Films 491 (2005) 1-17



feedback control