



## Ion Beam Sputtering for Dense Oxide Coatings

Authors: David Sanchez and Samuel Pellicori Materion Coating Materials News

High-energy deposition processes produce desirable film qualities that provide high transmission, stability to external environmental influences, resistance to laser damage by high-energy lasers, and low stress. These processes rely on the creation of high-energy ions and plasmas to provide the kinetic energy and reactivity necessary to deposit dense film layers. By way of comparison of process energies, the energy provided by resistance-heated evaporation is a few 10ths eV. High substrate temperature ~300 °C is required with resistance-heated evaporation to supply kinetic energy to mobilize the arriving adatoms and produce moderately dense microstructures. When augmented by simultaneous heavy-ion bombardment (Ar+) with energies of ~50-70 eV in the process called Ion Assisted Deposition (IAD), the thermal films can be compacted to an amorphous microstructure of higher densities. More complete oxidation also results in the process in which reactive oxygen ions are also produced. The results are films that meet the already mentioned favored qualities to varying degrees.

However, regarding properties such as internal stress, optical absorption and environmental stability, the process leaves much to be desired. More energetic processes, such as magnetron sputtering with energies is in the range ~10 eV, produce repeatable high quality films with low stress. Depending on the process parameters associated with the variety of techniques, other preferred properties may vary in quality. The user must adapt process parameters to fit the materials and application.

Ion beam sputtering (IBS) was developed to enhance these properties: environmental stability, laser damage threshold and optical absorption. IBS today produces the highest quality oxide-compound films. In the process, ions of energy ~1kV from a Kauffman-type ion source sputter material from a target, and the high-energy Ar+ and  $O_2$  ions bombard the growing film. IBS is regarded as a reactive process from a metal, alloy or even a full oxide target. The high energies present result in stoichiometric amorphous films with near bulk density, low scatter, and near zero absorption at lower substrate temperatures. Applications requiring the minimum absorption, such as ring laser gyroscopes and high-energy laser coatings, benefit the most from IBS. The high packing density of the films ensures that the spectral properties of IBS coatings are invariant between humid and arid environments. Currently, metal oxides dominate IBS since the reactive gas is well understood and is also non-hazardous and transparent in the critical UV, VIS and NIR ranges. Fluorides, Nitrides and Carbides can be created, but the reactive gases or the mechanical stability of the target, can pose significant challenges.

One aspect of high packing density that can become an issue is the high stress that is built into thick coatings by the high compaction energy of the impacting ions. Measures are implemented to minimize this potential problem by adjusting deposition parameters. Large areas can be uniformly coated by IBS, but stress limits the number of thick layers possible. While the substrate temperatures can be lower than IAD with electron beam processing (eBeam), the targets themselves are subject to much higher heat flux as a function of incident sputtering lon Beam angle, intensity and interference.



The higher target temperature is not completely negated by advanced target cooling approaches such as rolling and annealing. Greater detail will follow concerning the risks associated with forged microstructural variations in metal targets or fragile brittle metals and the limitations of pressed compounds.

*Figure 1* shows two basic arrangements of a single beam IBS. On the left, the substrates and targets are mounted on their respective turrets to permit multiple materials to be sputtered by selecting target material; also, to improve thickness uniformity by rotating the substrates in the sputtered beam. A second ion source bombards the growing film to increase packing density.



Figure 1. Common configurations of Ion Beam Sputtering systems. Left shows the basics of a dual ion beam system. Right is a configuration using a planetary substrate holder. (Patent: US 7871563 B2)

The right-hand figure shows the configuration that uses a planetary rotation for coating larger substrates or many smaller ones. The targets and target assemblies can be cooled using a rotating feedthrough in the chamber wall and/or water-cooled backing plates. Not included in the figures are the all-important gas inlets, the optical and crystal thickness rates and thickness monitoring accessories. Variations might include multiple ion sources to increase deposition rate, enhance surface diffusion and optimize film density and composition. With the growth of NIR lasers in manufacturing for optics and laser crystals, great strides have been made to increase the rate and stress of advanced IBS processes.

Because high energies are involved, IBS produces film with environmentally stable and superior optical properties. The ion energies created by the sputter source are higher than those from magnetron sputtering although layer growth rate is lower. The arriving adatoms have high energy of mobility; as a consequence they find and fill vacancies in the growing film layer to produce maximum packing density. Another consequence is that crystal growth is discouraged and amorphous microstructure results. That microstructure has fewer defects, complete oxide stoichiometry, and a smoother surface. IBS films are



famous for low scatter and low absorption losses measured in a few parts per million. Their laser damage threshold is also very high.

IBS is a reactive process that uses metal oxide compounds for coatings used in wavelength ranges from ~300 nm to ~1500 nm. Substrates remain at low temperatures. Deposition rates are as low as 1/10 that of eBeam deposition. The slow and highly reproducible deposition rate permits accurate and precise monitoring that yields control of the layer composition and thickness. These characteristics promote process automation.

# Preparation of Sputter Targets for IBS

Typical targets are composed of Tantalum (Ta), Niobium (Nb), Silicon (Si), and Hafnium (Hf) and sputtered reactively to deposit films of their oxides for optical applications. The primary difference between magnetron and IBS targets is the degree and severity of the rolling/annealing steps required. The additional processing increases cost and in the case of Ta, sometimes risks overworking the target which may erode unevenly or warp in-situ. The metal ingots are first cast then hot rolled and annealed (forged) to refine the grain structure. While IBS is generally more forgiving than magnetron sputtering with large grains or gaps, IBS is more sensitive to grain orientation. When Tantalum experiences more forging, it is notorious for developing bands of different target grain orientations. Therefore, great care is required to produce a consistent homogeneous and regular-grained Tantalum IBS target structure.

Niobium tends to exhibit more forge residual stress than other metals and can warp in-situ due to the higher temperature. Silicon and Germanium can be single or poly-crystalline and may introduce some risk to the process.  $SiO_2$  plates that are polished on both sides may have difficulty in adherence or simply be cosmetically inferior for bonding due to high transparency. Precious or expensive materials can take advantage of advanced bonding techniques such as <u>Materion's Patented modular insert</u> technology [1] which minimizes the spend on new material.

One additional note on target material preparation: when a material cannot be rolled, for example, a brittle alloy or metal, and cannot be grown/sliced or formed (glass), it must be pressed (cold or hot). Pressed targets are generally less desirable for IBS due to chipping or cracking issues, plus cold pressing of powder introduces more challenges with binders or density variation. All powder targets risk particle formation and void content, therefore they further limit sputtering rate. IBS processes also run very hot compared to magnetron, so debonding is a risk with even cooled metal targets (indium melt). Water-cooled backing plates are used but are more expensive to maintain and require great care during debonding/rebonding of replacement targets. This is especially critical for modular or mosaic approaches for expensive materials or gross alloys.

Ion Beam Sputtering is becoming a popular deposition process for metal oxide coatings for near-UV to near-IR coatings because of the high degree of control of thickness and composition that the process provides. The films are low scattering, have high environmental stability, and high laser damage thresholds.



# **More About Sputtering**

Learn more about <u>thin film coating material</u> and sputtering processes from Materion Advanced Materials, an industry leader in supplying <u>specialty precious and non-precious materials</u> for a range of applications.

# Reference:

[1] Figure 1 - https://www.google.com/patents/US7871563

# Authors:

David Sanchez, Sr. Materials/Applications Scientist Materion Advanced Materials, Milwaukee, WI Email: <u>David.Sanchez@Materion.com</u>

Samuel Pellicori, Consultant Pellicori Optical Consulting, Santa Barbara, CA Phone: 805.682.1922, Email: <u>pellopt@cox.net</u>

Material Advanced Materials is a global supplier of premier specialty materials and services. Our offerings include precious and non-precious thin film deposition materials, inorganic chemicals and microelectronic packaging products. In addition, we offer related services to meet our customers' requirements for precision parts cleaning, precious and valuable metal reclamation and R&D. We support diverse industries including LED, semiconductor, data storage, optical coatings, large area glass and aerospace.