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Complete coating of metal rings by ion beam sputtering of a W-shaped concave target with a broad-beam ion source

Sevda Ayata [a,*], Koumei Baba [b], Wolfgang Ensinger [c]

[a] Chemistry Department, Faculty of Science, Dokuz Eylul University, 35160 Izmir, Turkey
[b] Industrial Technology Center of Nagasaki, Omura, Nagasaki 856-0026, Japan
[c] Department of Materials Science, Darmstadt University of Technology, 64287 Darmstadt, Germany

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ABSTRACT

Ion beam sputter coating with the substrate at ambient temperature or water-cooled is a well-known process for coating temperature-sensitive substrates with films of high quality, despite the low process temperature. However, ion beam based methods suffer from an intrinsic drawback, the so-called *line-of-sight* restriction. Since a directed beam of the material to be deposited is used, only that part of a sample that is "seen" by the particle source is properly treated. This renders coating of 3-dimensional objects with ion beam methods difficult. Particularly challenging is to completely coat such objects from all sides. A typical example are rings. When they are to be treated with ion beam techniques, sophisticated manipulation of substrate or ion beam or even both is required. Despite these problems, under certain circumstances it is nevertheless possible to apply ion beam techniques for treating/coating 3-D objects. When ion beam sputter coating is used, the sputter target, i.e. the source of the material to be deposited, is usually flat. With such a sputter target, a ring can hardly be coated uniformly. However, when the sputter target is formed according to the substrate shape, here as a concave/convex double-cone, a very effective coating can be achieved. This is demonstrated by coating rings for corrosion protection.

1. Introduction

Deposition of microstructurally dense coatings by physical vapour deposition methods, such as electron beam evaporation and plasma-based sputtering, usually requires an elevated substrate temperature [1,2]. In contrast, with *ion beam assisted deposition* and *ion beam sputter coating*, the assist ions transfer enough kinetic energy into the growing film for a favorable structural film growth [3,4]. Thus, temperature-sensitive substrates can also be treated. However, there is a basic drawback of ion beam methods: they are *line-of-sight* techniques. A directed beam of ions does only hit the sample to be treated when the latter is in direct sight contact to the ion source. For this reason, ion beam based coating of three-dimensional objects is difficult. Particularly difficult is a complete hermetic coating of such objects. In this case, the workpiece and/or the ion beam has to be manipulated in a complicated way. Here, plasma based processes running at higher gas-pressure with coating "round the corner" by atomic and molecular collisions are more favorable. In order to be able to make use of the advantages of ion beam methods with respect to temperature-sensitive materials, an ion beam sputter deposition process has been developed that does not use the usual flat sputter target, but one which is shaped according to the dimensions of the substrate to be coated. In the following, a method is described which allows to coat metallic rings completely and thus protect them from corrosion.

Among the heat-sensitive metallic alloys to be coated, several are particularly prone to corrosion. Tempered 7075 aluminum alloy, used e.g. in automotive and aerospace applications, with a composition with roughly 5–6% zinc, 2% magnesium, and 1–2% copper shows a much higher strength-to-density ratio, but a considerably lower corrosion resistance than Al. Steel AISI 52100 is a high carbon, chromium containing low alloy steel for e.g. ball bearings with around 1% Cr and 1–2% C. It can be tempered to high hardness values, but suffers from corrosion in aqueous environment due to the presence of carbon. Both alloys require corrosion protection, but do not tolerate high treatment temperatures. Rings of these materials were coated with Cr and Al with the before-mentioned method and were subjected to corrosion and film adhesion tests.

2. Experimental details

2.1. Set-up of the ion beam coating apparatus

The apparatus, shown in Fig. 1, consists of a cubic vacuum chamber. A base pressure of 10^{-5} Pa is maintained by a turbomolecular pump combined with a rotary fore-pump. Horizontally, a multi-aperture radiofrequency ion source operating at 13.56 MHz is mounted. It has a beam aperture of 200 mm diameter and a maximum acceleration

* Corresponding author.
E-mail address: sevda.ayata@deu.edu.tr (S. Ayata).

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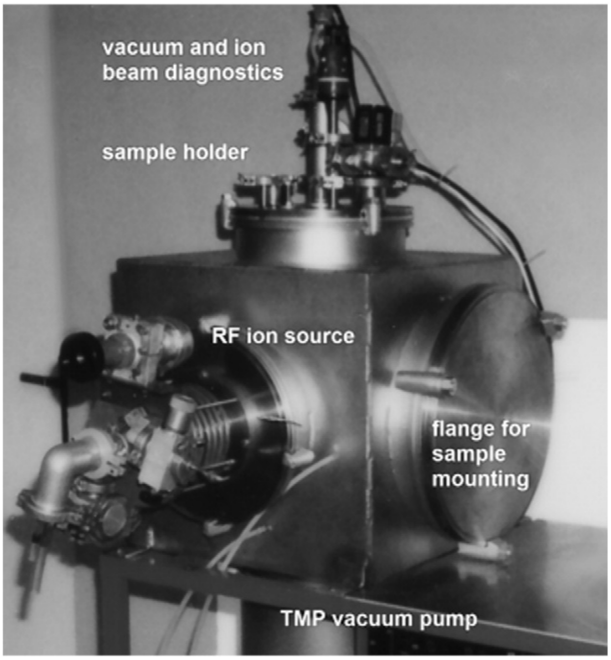

Fig. 1. Photograph of apparatus for ion beam sputter coating of rings, with cubic vacuum chamber, radio frequency ion source and target/substrate holder.

voltage of 10 kV with a maximum ion beam current of several mA. The beam can be cut off by means of an electrically movable shutter with an integrated Faraday cup between the ion source and the sputter target.

The sputter target is composed of a concave cone with an outer diameter of 200 mm and a convex cone tip with a diameter of 100 mm inside the concave part. In cross-section, the sputter target has the shape of the letter W. The cone angles are 45° so that in all positions the ions impinge at an angle of 45° when the ion beam is symmetrically directed towards the sputter target. Atoms are essentially sputtered in all directions, however, with a preferential forward direction. The conical sputter targets are mounted on a water-cooled copper target holder to avoid excessive heating.

The ring-shaped substrate holder is symmetrically mounted inside the sputter target. It is cooled by means of a water pipe. The closest distance between the sputter target and the ring substrate is 2 to 3 cm, depending on its position within the sputter target. Fig. 2 shows a schematic presentation of the set-up.

2.2. Experimental parameters

AISI 52100 steel and 7075 Al alloy rings with a central diameter of 100 mm and a thickness of 8 mm were degreased ultrasonically with acetone and isopropanol and washed with distilled water, before they were mounted on the sample holder ring. A sputter target of an Al sheet (99.9% purity) on a double conical copper carrier and another one of stainless steel AISI 304, galvanically coated with 100 μm Cr were used. Each ring was treated in two steps; first, it was coated from one side, thereafter it was turned around and then coated from the second side. The process is schematically shown in Fig. 3. For the deposition, the ion source was run with argon gas at an extraction voltage of 10 kV with a current of 820 μA. The operational gas pressure was 10^{-2} Pa, due to gas release from the ion source. At this pressure, the mean free path of all involved species is larger than the distance between ion source and sputter target (20 cm), and between sputter target and substrate. As an example, the mean free path of nitrogen molecules at room temperature is around 60 cm at 10^{-2} Pa. The film thickness was estimated from surface profiler (Dektak) measurements at edges of shielded parts. The adhesion was tested with the pin pull test. Each ring was cut in eight equal segments. Pins were glued to it with epoxy glue and were pulled off perpendicularly. The pull-off force at adhesive failure was recorded. The upper limit of the test is around 80 MPa, when the glue fails cohesively.

Electrochemical corrosion tests with potentiodynamic current-vs-potential measurements were carried out in a standard three-electrode set-up with Standard Calomel Electrode (SCE) as reference electrode and a platinized Titanium plate with a diameter of 100 mm as counter electrode. The corrosive solution for the steel was 1 M acetic acid buffered with sodium acetate to pH 5.6. The Al alloy was tested in 0.1 M NaCl solution. The temperature was kept at 25 °C. The electrical contact was established by means of a screw that was covered with an inert resin in order to avoid contact with the corrosive solution. The rings were completely immersed in the corrosive solution in a thermostated 1 L glass beaker. The potential scan rate was 0.01 and 0.001 V s^{-1}, resp. The measurements were run with a computer-controlled potentiostat.

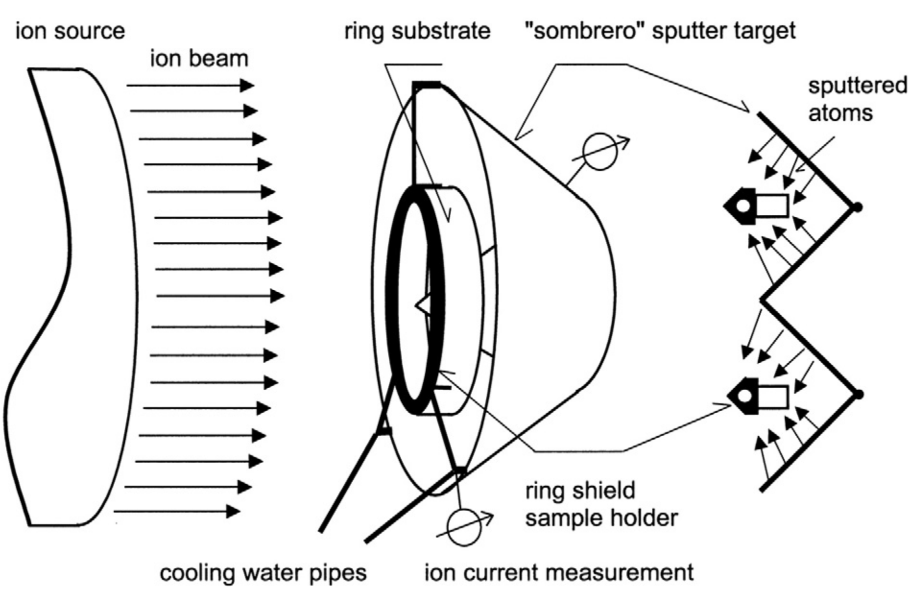

Fig. 2. Schematic presentation of set-up for coating rings by sputtering a double-conical (W-shaped) sputter target with a broad-beam ion source.