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Thin Solid Films 471 (2005) 170–176

www.elsevier.com/locate/tsf

A modified blister test to study the adhesion of thin coatings based on local helium ion implantation

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Received 23 September 2003; received in revised form 19 May 2004; accepted 18 June 2004
Available online 3 August 2004

Abstract

A modified blister test has been developed based on helium ion implantation into selected areas of the metal substrate prior to the coating deposition. After a post-deposition thermal annealing, blisters are formed by agglomeration of the implanted gas at the ceramic–metal interface. This method can be used to control the pressure in the blister which eventually may lead to delamination at the periphery of the blister. A microsieve with a regular array of circular holes is used during the implantation to assure the initial blister size. Two different microsieves were employed in this work, with pore diameters of 1.5 and 4.5 μm, respectively. The distance between the centres of neighbour pores is twice the pore diameter. Scanning Electron Microscopy (SEM) and Confocal Scanning Optical Microscopy (CSOM) observations allowed the determination of the blistering parameters such as the radius, the height and the blister volume. From the gas content and these parameters, the work of adhesion or energy release rate can be obtained.

In this work, we present the first results of this blister test applied to W–C:H films and multilayers of Ti and Al deposited by Physical Vapour Deposition on polycrystalline copper substrates. The copper substrates were implanted with 34 keV He^+ ions up to fluences of 3 and 5×10^{16} cm^{-2} before the deposition of the coatings and annealed afterwards in vacuum at temperatures from 773 to 1073 K for 30 min. Delamination of the Ti/Al multilayer coatings was already detected after annealing at 873 K with an energy release rate estimated to be 0.5 J m^{-2} at a typical helium pressure of 10^7 Pa. No delamination but only helium swelling was observed for W–C:H coatings annealed at 1073 K. Results of experiments on uncoated copper samples are also shown in order to explain the mechanism of helium bubble growth and helium release that causes the creation of the blisters.

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PACS: 68.35.Gy; 62.20.-x; 61.16.Bg; 85.40.R
Keywords: Adhesion; Ion implantation; Ceramics; Metals

1. Introduction

Coatings on materials have numerous applications, which vary from improvement of wear resistance to embellishment of the surface of objects. In coating technology, considerable attention has been paid to reliable ways of measuring the adhesion of coatings to substrates. One standard test has been the blister test, originally proposed in 1961 by Dannenberg [1], involving the injection of a liquid between a substrate and coating in such a way that the coating is detached in the form of a blister. In 1969, Williams [2] introduced the well-known blister test in which gas pressure was built up by feeding gas through a circular hole at the backside of the substrate. He applied this method to measure the fracture energy of thin coatings on rigid substrates. A constrained blister geometry was introduced by Napolitano et al. [3] to avoid uncontrolled growth of the blister at the liquid or gas pressure. This was

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doi:10.1016/j.tsf.2004.06.129

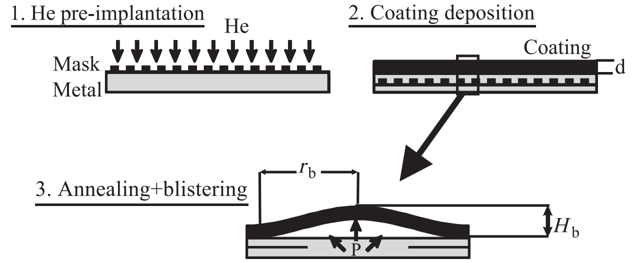

Fig. 1. Schematic representation of the modified blister test proposed. In step 1, the helium ions are implanted in the metal substrate through a mask prior to the coating deposition (step 2). After annealing, the gas is promoted to the interface and causes blistering at the implanted areas by exerting a pressure P to the coating. The radius (r_b) and the height (H_b) of the blisters are related to the adhesion properties of the coatings.

used, among others, by Lai and Dillard [4,5]. The release of gas implanted into the substrate through a polymer film for generating blisters on materials covered with thin films was proposed by Borisenko et al. [6] in 1995. They showed a correlation between gas blister parameters and the adhesion properties of hydrogen-implanted thin silver films on glass substrates. An overview of the theory describing the blister tests is given by Williams [7]. Assuming that the blister lid behaves elastically, the energy release rate (G) in the case of axisymmetric membranes is given by,

$$G = cP_dH_d \tag{1}$$

with c as a geometrical constant ~1, P_d as the pressure and H_d as the height of the blister when delamination occurs.

Against this background, this paper examines the possibility of using gas release from substrates previously implanted with helium to develop an improved blister test to measure the adhesion of the thin coating to the metal substrate. The method aims at improved control over the blister by confining the gas only to a well-defined area. In Fig. 1, the different steps taken during the test are illustrated:

1. Helium ions are implanted in the sample substrate through the holes of a mask;
2. The coating is deposited on the substrate; and
3. By thermal annealing, gas that accumulates at the coating/metal interface is released.

The low permeation of the gas through the coating will lead to the formation of blisters at the interface. The shape and the geometric parameters (height and radius) of the blisters can be measured by means of Confocal Scanning Optical Microscopy (CSOM). They can be used to determine the energy release rate when delamination occurs.

2. Experimental details

In order to test the method, two uncoated polycrystalline copper samples were prepared. Before the implantation, the polycrystalline copper samples were stretched 2% prior to polishing the surface to a roughness lower than 1 µm. Subsequently, they were annealed to remove defects in a vacuum of 10^{-5} Pa at 923 K for 30 min in the presence of hydrogen. The localised gas implantation was performed using a VARIAN 350D ion implanter at 34 keV with fluences of 3 and 3.5×10^{16} He ions/cm^2. The implantations were performed through microsieves with a regular array of pores provided by AquaMarijn Micro Filtration. In Fig. 2, the layout is shown of the copper sample with the location of the microsieve. Two different pore sizes were used for the implantations: 1.5 and 4.5 µm. The distance between the centres of neighbour pores was twice the pore size. As the microsieve partly covered the samples, two implantation zones were considered. These will be referred hereafter as the direct implantation area and microsieve implantation area, respectively (see Fig. 2). The helium implantation depth was 129±57 nm as calculated by TRIM [8]. After the implantation, the samples were heated in a typical vacuum of 10^{-5} Pa to 973 K for 30 min in order to promote the helium ions to surface. Once the copper surface is coated, all this helium will be collected at the interface.

W–C:H films and multilayers of Ti and Al were deposited by Physical Vapour Deposition on polycrystalline copper substrates prepared in a similar way as described above. The W–C:H films were deposited using unbalanced magnetron sputtering in an argon/acetylene atmosphere in a Hauzer HTC-1000 production scale coating system, as described by Strondl et al. in Refs. [9] and [10]. The films have a thickness of 2 µm with an adhesive intermediate chromium layer of 200 nm. The Ti/Al multilayer coatings were deposited at the Universidade de Coimbra (Portugal) from pure Ti and Al targets (99.99%) by d.c. reactive magnetron sputtering. The discharge power applied to Ti and Al targets was 990 and 500 W, respectively, in order to get approximately the same thickness in the individual layers of the coating. The targets with 150×150 mm dimensions were vertically placed at 90° and the substrate holder in the centre of the deposition chamber was rotating. A negative bias voltage of 70 V was applied to the substrate during the deposition and the Ar pressure was 3×10^{-3} Pa. The substrate surfaces were ion-cleaned with an ion gun before coating deposition. The

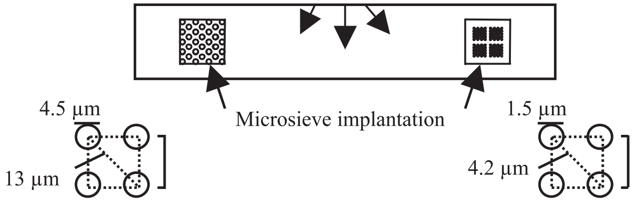

Fig. 2. Top view of the layout of the sample used during implantation. Indicated are the direct and microsieve implantation areas. A closeup of the microsieve areas shows the two different pore sizes (1.5 and 4.5 µm) and periodical array of holes. In the bottom, two confocal pictures show the periodical pattern of the AquaMarijn microsieves with (a) 1.5- and (b) 4.5-µm pore diameters used in the experiments.

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