Vacuum 83 (2009) S186-S189

Contents lists available at ScienceDirect

Vacuum

journal homepage: www.elsevier.com/locate/vacuum

Properties of (Ti,Cr)N and (Al,Cr)N thin films prepared by ion beam assisted deposition

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A R T I C L E I N F O

Article history: Received 16 June 2008 Received in revised form 22 January 2009 Accepted 30 January 2009

Keywords: Ternary compounds IBAD Surface topography Bubbles Tribology

ABSTRACT

(Ti,Cr)N and (Al,Cr)N thin films were prepared by nitrogen ion beam assisted deposition of Cr, Ti or Al (IBAD) atoms on a titanium, chromium or aluminium substrate. In this way, layers featuring a diverse composition were obtained. The actual profile of the implanted and deposit atoms differed from the theoretically assumed process. Some of the implanted nitrogen formed gas bubbles under the sample's surface. The gas bubbles released the nitrogen upwards while the local temperature increased during the implantation or during a later heating process. Craters are formed from the bubbles, which were clearly visible on the AFM photographs. Annealing the modified surfaces in a vacuum always changed the surface topography of the sample. The lowest wear rate and the lowest friction coefficient were observed in the Cr–Ti–N and Cr–Al–N samples.

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1. Introduction

(Ti,Cr)N and (Al,Cr)N thin films are intensively tested in terms of very high tribological properties. They belong to the third generation anti-wear coatings prepared with the PVD (physical vapour deposition) methods [1,2]. However, the PVD methods require the substrate temperature to be above 200–250 °C to obtain good adherence of the layer to the ground and to obtain a high hardness value. These high-substrate temperatures inhibit the use of TiN, CrN and AlN coatings in many applications where the substrates (e.g. pre-stressed tools) or substrate materials (e.g. plastics, compounds) cannot withstand such high temperatures.

The ion implantation method is a universal method for creating thin alloy layers. The method does not have any of the abovementioned limitations and can be used in the wide temperature range of the implanted sample: from helium temperatures to several hundred °C. The nitrogen implantation has been frequently used to improve the mechanical properties of the surface layers of aluminium and titanium [3,4].

It was interesting to check how the ternary compounds (Ti,Cr)N and (Al,Cr)N can be obtained with the nitrogen implantation method combined with aluminium, titanium and chromium

* Corresponding author. E-mail address: p.budzynski@pollub.pl (P. Budzynski). deposition, and if they are stable at a temperature similar to the local working temperature during dry friction, i.e. 450 °C. We were interested in the surface topography and crystalline structure of (Ti,Cr)N and (Al,Cr)N thin films produced with the IBAD method.

2. Experiments

(Ti,Cr)N thin films were obtained in two ways. Chromium was deposited on a titanium substrate in cycles (alternately) and nitrogen was implanted – such a sample is marked as <u>Ti</u>–Cr–N. The second method of creating (Ti,Cr)N thin films is based on a cyclic Ti deposition on a chromium substrate and alternate nitrogen implantation – the sample is marked as <u>Cr</u>–Ti–N. The (Al,Cr)N thin films were obtained in similar manner and the obtained samples were marked <u>Al</u>–Cr–N and <u>Cr</u>–Al–N. Additionally, we deposited aluminium on the aluminium substrate – the Al–Al–N sample.

The deposited Al, Cr or Ti atoms came from high-clean metallic discs sputtered with nitrogen atoms. The energy of the implanted (also sputtering) nitrogen ions was 60 keV. Implantation was alternately performed with sputtering (IBAD): sputtering, implantation, sputtering, etc. The whole process ended when the total fluence of nitrogen atoms used in sputtering equalled the fluence of the implanted nitrogen atoms, and each of them amounted to 5×10^{17} N⁺/cm². During the deposition of layers by sputtering and implantation, the samples had thermal contact with the handle cooled with flowing water.



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The sample surface topography was tested with an atomic force microscope (AFM). The AFM microphotography was prepared with the use of the VSXM programme [5]. All samples show the columnar structure. Figs. 1 and 2 show the surface of the samples just after the implantation. The same structure was also observed in the samples obtained with different methods, e.g. for the Cr-Ti-N coatings deposited in a medium frequency magnetron sputtering system on Si (111) substrates [6]. After annealing, the craters occur on the surface of the tested layers. The craters are seen well particularly on the surface of the Al-Cr-N sample. According to the surface profiles obtained with the AFM (atomic force microscopy) method, the craters are discovered about 30 nm deep, which is equal to $\sim 1/3$ of the sedimentation layer thickness. The craters are the remains of the gas bubbles where the nitrogen was accumulating during the implantation. Similar craters were observed in the (Ti,Al)N thin films [7] and in aluminium after the nitrogen ions implantation with a fluence of 1.6×10^{16} ions/cm² [8]. However, the authors of the research [8] assumed that the hollows emerged as a result of cracking caused by tension during the implantation.

The projected distribution of implanted and deposited atoms in a titanium, chromium and aluminium sample was calculated by a modified Satval programme [9]. The results of the performed calculations for <u>Cr</u>-Al–N and <u>Cr</u>–Ti–N are shown in Fig. 3. The rectangle marks the total thickness of an aluminium or titanium layer deposited on the substrates during sputtering.

The experimental profiles of the implanted ions and the deposited atoms were measured with the SIMS (secondary ion mass spectroscopy) method. Experimental profiles usually differ

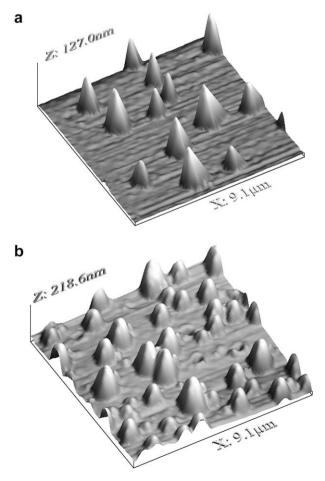


Fig. 1. The <u>Al</u>-Cr-N sample surface: (a) as implanted, (b) after annealing in vacuum at a temperature of 450 °C during 48 h.

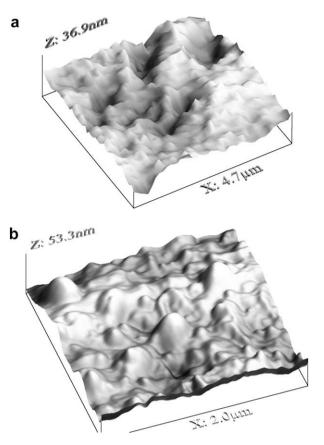


Fig. 2. The sample surface as implanted: (a) Cr-Ti-N, (b) Cr-Al-N.

from the theoretical calculations due to, among others, atoms' interactions: Ti, Cr, and N, which create chemical compounds that are not taken into consideration in the computer simulations. After annealing, the nitrogen in the \sim 120 nm thick upper layer has significantly decreased, which was mainly due to a decay of the gas bubbles inside the <u>Al</u>-Cr-N and <u>Ti</u>-Cr-N layer. Additionally, some nitrogen atoms from the layers located closer to the surface diffuse deep down into the sample. The chromium atoms also diffuse deep into the titanium sample. The chromium atoms' diffusion process is simpler due their smaller sizes compared to the parent atom of the sample (titanium).

The crystallographic structure and chemical composition was investigated by GXRD (grazing incidence X-ray diffraction) at angles of 0.5, 1, 2, and 3° of the primary beam (CuK_{α} radiation). The change of peak intensiveness at various X-ray angles was used for determining the relative phase depth location that the peaks came from. The GXRD measurements showed that the obtained ternary (Al.Cr)N thin films had a biphasic structure. The cubic phase $(Al_{1-x}Cr_x)N$, $x \sim 0.5$ is discovered in the surface layer. Under this layer, directly near the aluminium there is a c-AlN phase, and the CrN layer is on the chromium base. Annealing did not change the phase composition of the surface layer. The changes occur in the layers that are in direct contact with the metal substrate. A part of the metastable AIN cubic phase has transformed in the hexagonal phase. And the CrN phase has transformed in the Cr₂N phase. The phase structure of the (Ti,Cr)N thin films is similar. The cubic phase $(Ti_{1-x}Cr_x)N$ of $x \sim 0.5$ (a = 0.417 nm) was dominating on the surface. Directly near the metal layer of the substrate (i.e. deeper) in the Ti-Cr-N sample there is a TiN layer, and in the Cr-Ti-N sample there is the CrN layer. Annealing almost did not change the phase composition of the Ti-Cr-N sample. And in the Cr-Ti-N sample, a part of the CrN phase has transformed in the Cr₂N phase after annealing.

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