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Evaluation of residual stress and adhesion of Ti and TiN PVD films by laser spallation technique

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ABSTRACT

The laser spallation technique was applied for measurement of residual stress and adhesion of thin films. Two films of different properties, ductile and soft Ti, and hard and brittle TiN, were studied. The films were produced on 304 steel substrate by PVD method. The residual stress value obtained by laser spallation technique LST were compared with stress value from X-ray diffraction method. Good agreement of stress values measured by both methods was attained. Additionally, the interface strength of the films was tested by laser adhesion spallation technique LASAT with use of VISAR system. It was shown that shock wave induced by a nanosecond laser pulse adequately determines properties of PVD thin films on metal substrate.

1. Introduction

The shock waves create many unique possibilities in materials engineering although evaluation of properties of materials and layers undergoing high speed deformation is a serious experimental issue [1,2]. There are several methods for material testing at high speed deformation, such as the Split Hopkinson Pressure Bar (SHPB) method, miniaturized direct impact test, shock and explosive methods, however all these tests are complex and destructive [2]. Mechanical properties at high strain rate may be tested also by means of dynamic hardness testers. Strain rate in these devices is about $10^3\ s^{-1}$. Dynamic-plastic tests are applied the most commonly by pressing the indenter with the Poldi's hammer while in dynamic-elastic test (Shore method) a bounce of the indenter or spring is measured [3]. Recently, the reports on development of new methods for measuring the dynamic hardness using a high velocity gas gun have been elaborated. The strain rate in these methods is about 1500–2200 s^{-1} [3,4].

In 60-ties the high-energy laser pulse was applied as pressure load [5]. The short laser pulse interaction with material generates plasma and a pressure wave. The shock waves with high amplitudes, causing compression stress exceeding yield point of metals can be obtained [6] for surface covered with inertial layer, transparent for the laser beam (confined regime) [7,8]. Nanosecond high power pulses and carefully selected absorption layer as well as inertial layers, allow generating a pressure wave from a few to several GPa [9].

The use of short laser pulses in order to generate high-pressure shock waves create many unique possibilities in materials testing. Contrary to collision systems, wider range of pressures, speed and deformation settings may be achieved as a result of changing a shape and time duration of the laser pulse. On the basis of the laser shock waves the new diagnostic methods of dynamic behaviour of material and layer [10], as well as adhesion of thin films could be developed [11].

Thin films are an important component of many microelectronic, optical and micromechanical systems as well as cutting tool coatings. During their manufacture a large amount of a residual stress is induced, that has significant influence on their mechanical properties and overall efficiency. In certain conditions, the residual stress may cause layer delamination from substrate or its cracking. The most well-known practical techniques of measuring the adhesion of thin layers are scratch, peel, pull, blister or indentation test. Laser Spallation Technique LST was first introduced by Vossen [12]. In this method a layer is loaded with the stress wave created by short laser pulses. Accompanied phenomena were wider described in [13–19], and the technique was called LASAT (Laser Shock

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Adhesion Test). Adhesion of films was examined by the LASAT in many systems, such as components of electronic devices [13], PVD and CVD coatings in tool applications [20], plasma layers [21], joined materials or composites [22]. Mostly the interface strength of thin films few micrometers thick, on ceramic substrate, was analyzed [11–16].

In the 1990s, attempts to use the pressure wave generated by a laser pulse for testing the adhesion of thin layers, obtained by PVD and CVD methods, were made. Tensile stress at the interface of the material/layer phases, which caused separation of the layer from the surface, have been studied [18]. In these methods adhesion of layers is calculated on the basis of a speed of the layer. The accuracy of the method depends mainly on the accuracy of measuring of velocity of the back sample's surface. Due to a very short time of the process, from several to tens of nanoseconds, highly advanced measurement techniques are required [18,19]. A number of review articles on the LASAT measurement methods are published for specific applications and layer materials. Among others they refer to TiN layers [15,20], hydroxyapatite [22], thermal barrier coatings EB-PVD TBC with the use of shock wave propagation in two dimensions (LASAT 2D) [23] or carbon fibre reinforced composite CFRP [24]. Also the surface shapes of the substrate as well as configuration of samples are profoundly analysed [24,25].

Based on laser spallation technique new method of residual stress measurement was proposed by Ikeda at el. [26]. During delamination of thin films, a protuberance occurs in films that have compressive residual stresses [27,28]. In many thin films made by PVD and CVD methods compressive residual stress generate. The film and substrate are made of materials that has different thermal expansion coefficient thus during a cooling the film gets residual stress. To release this compression the film tends to fit off the substrate and fracture of interface can take place. Residual stress can be determined from a shape of protuberance without knowing elastic properties of film. Analytical solution for a pressurized membrane was applied taking into account the pressure and further neglecting residual stress release by the elastic deformation [26]. The advantage of this method compared to the X-ray diffraction is possibility of stress measuring of the film that has strong texture or poor crystallinity. In such case a conventional X-ray method cannot be used.

The paper presents experimental results of residual stress measuring by a laser spallation technique. The technological thin films TiN and Ti, produced by PVD method on steel substrate, were studied. The residual stress value determined by the laser spallation technique were compared with stress value obtained by X-ray diffraction methods. The adhesion strength was also determined by LASAT method with use VISAR system. It was shown that shock wave induced by nanoseconds laser pulse can be suitable tool for determination of properties of technological thin films.

2. Experimental method

The study of residual stress and adhesion of PVD thin films by laser spallation technique was carried out for typical commercial metals and thin films. As a substrate a stainless steel, EN X5CrNi18-10 1.4301 (304), was used. Two kinds of commercial thin films, TiN and Ti, deposited by PVD method in Surftech manufacture [29], were tested. Samples were prepared before the deposition process. Round samples of a diameter of 10 mm were cut by WEDM method out of sheets 0.5 mm thick. Surface of material was electrolytic polished and cleaned before the deposition. Multi-step degreasing was applied: first in tetra-chloroethyl activated by ultrasound, then in aqueous solution of a detergent with a rinse in water, afterwards cleaning in alcohol and acetone activated by ultrasound, finally in vapor of tetra-chloroethyl.

The PVD process consists of the following steps: vacuum generation, heating up to 450 K, ion etching, coating deposition and cooling.

Nd:YAG Quantel YG 981E laser with a wavelength of 1.064 μm and pulse duration of 10 ns was applied for testing. The beam diameter was 2 mm. A diagram of the measurement system is shown in Fig. 1. The laser pulse (1) is directed through a glass (2) to the absorption layer (3) causing its evaporation and plasma generation. A pressure wave (4) is formed as a result of rapid expansion of a plasma plume and propagates into material (5). Graphite 5 μm thick was used as the absorption layer, while glass 1 mm thick was the inertial layer. At very short, nanosecond laser pulses, and a suitably selected type and thickness of the absorption layer, the thermal effects associated with the interaction between the beam and material are negligible [10,30]. This allows to examine such a case as pure mechanical interaction of the pressure wave with tested material.

Different pulse energy levels and thicknesses of substrate were applied to obtain proper conditions for delamination of technological layers. The values of pulse energy were: 0.5, 0.7, 1.0 and 1.25 J while substrate thickness had three values: 1, 0.8 and 0.5 mm. Table 1 shows test parameters and presence of films delamination.

In order to determine a strength of the interface a surface velocity was measured by a VISAR system. The studies were conducted at pulse energy 1.2 J for both films. Three thickness values of the steel substrate were applied. The parameters applied in the LASAT are denoted by the "⁎" mark in Table 1.

The VISAR (Velocity Interferometer System for Any Reflector) determines the velocity of moving surface by measuring the Doppler shift of laser light reflected from the surface. It is sensitive to wavelength; therefore, it transforms changes in the wavelength to changes in the intensity of four output signals. Afterwards these intensities are converted by fast photodiodes to electrical signals recorded by an oscilloscope. Velocities can be determined in the range from m/s up to km/s and with sub-nanosecond time resolution with accuracy ±1%. The observed surface does not need to be mirror polished. Changes in its reflectivity or in the background light have no effect on derivation of velocity.

The properties of the films were controlled before testing in order to confirm the producer declaration. The following properties were measured: roughness, thickness and hardness. A surface geometrical structure of the films was studied on scanning profilometer. The roughness parameters Ra, Rz were determined according to ISO 4287:1997. For selected samples the metallographic cross-sections were made perpendicularly to the surface and thickness of the films was measured on a Scanning Electron Microscope SEM. A microhardness test of the films was carried out using the Vickers method at a load of 0.2 N (20 gf). The hardness values of

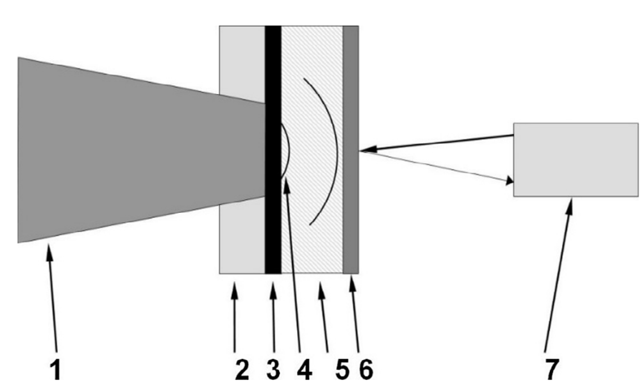

Fig. 1. Experimental scheme for testing of adhesion of thin films to the substrate: 1 – laser pulse; 2 – inert layer (glass); 3 – absorption layer (graphite); 4 – stress wave front; 5 – substrate; 6 – thin film; 7 – VISAR interferometer.