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Analysis of Titanium-Coated Glass and Imidex (PI) Laser Bonded Samples

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Based on previous results of bond strength, scanning electron microscopy(SEM)/energy dispersive spectroscopy (EDS) and x-ray photoelectron spectroscopy (for thin film thickness in the range of 50 to 200 nm range), it is expected for a moderate film thickness of titanium (over 50 nm) for the system of sputtered Ti-coated glass/polymer two factors play important roles in getting strong bond between Ti/Polyimide interface: (i) mechanical interlocking property and (ii) chemical bond formation such as Ti-C, Ti-O between Ti and imidex (PI) film. In this study, a systematic investigation has been conducted to understand the effects of thin films on bond quality and on failure mechanism of the interface between 400 nm sputtered Ti-coated glass/imidex (PI) system. This article basically studies if for this higher film thickness the failure pattern and bond strength are consistent with the previous data. From previous studies (for thin film thickness of 50 to 200 nm) the conclusion extracted is thin film with thickness of less than 50 nm exhibited low bond strength when compared to film thickness over 50 nm and from the results obtained in this study it is concluded that the bond reliability and failure modes of sputtered Ti film on glass are consistent even for a film thickness as high as 400 nm and three types of failure modes are found : (i) cohesive failure mode, (ii) Ti/glass interface failure mode, and (iii) glass failure mode. The roughness value for this coating thickness is 17 nm.

Keywords bond strength, chemical bond, cohesive failure mode, failure modes, mechanical interlocking property, scanning electron microscopy, x-ray photoelectron spectroscopy

1. Introduction

Our group has published a number of papers dealing with laser bonded micro-joints between titanium and polyimide and characterization of titanium/polyimide interfaces (Ref 1-7). Metal/polyimide interface has been a subject of intensive study due to its importance in microelectronic industry (Ref 8-11) and in aerospace application (Ref 12). Besides, metal/polymer system becoming a field of interest for researchers due to their potential application in the field of Bio-MEMS. Only a few metals such as titanium, cobalt, nickel, tantalum, and zirconium and noble metals such as gold (Ref 13), silver (Ref 14), platinum (Ref 15), and iridium (Ref 16) meet the requirements regarding corrosion resistance and non-toxicity in the human body. These metals show various levels of biocompatibility and are used for different applications either as pure metal or in the form of alloys. Pure titanium and titanium alloys show excellent bio-compatibility and used for dental and bone implants (Ref 17). Polyimide is chosen to use for intracortical neural implant (Ref 18) and nano-fluidic channels (Ref 19). Titanium could be used in conjugation with polyimide in a nano-fluidic device. Polyimide could be hermetically sealed by laser joining process on top of titanium-coated glass substrate; which has micromachined nano-fluidic channels on the surface. Those micro-pores effuse drugs in a controlled fashion.

Although our previous study (Ref 20) showed that titanium film with the thickness less than 50 nm produced poor bond strength compared to the thickness of titanium film in the range of 50 to 200 nm, it is very important to explore if performance of the system is consistent and reliable at very high film thickness as well. The film thickness of titanium used for this study is 400 nm. The scope of this study is to provide new insight into micro-scale precision laser joints for sputtered Ti-coated glass and polyimide film system as well as to represent the capability of such system for producing consistent bond strength and failure modes for thicker film of titanium.

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2. Experimental

2.1 Deposition

In this study, DC magnetron sputtering deposition method was used to prepare 400 nm sputtered titanium film on glass substrates. This system includes a deposition chamber, an ion source, a target, substrates, gas controller, and vacuum pump. Ar gas with a flow rate of 10 sccm was used to generate ions. The base pressure in the deposition chamber was of about 8 μTorr and input power was 50 W. Prior to deposition the substrates were chemically cleaned in an ultrasonic bath, separately with alconox solution, DI water, acetone, and methanol. The system was pre-sputtered for 15 min in vacuum by Ar ion bombardment.

2.2 Laser Micro-Joining

Laser transmission bonding (Ref 7, 21) is appropriate technique for bonding of dissimilar materials. A4-axis high precision laser motion system that manipulates the samples under the stationary beam was used to prepare the samples. This system provides a positioning accuracy of ±1 μm. A Nd:YAG fiber laser (λ = 1100 nm) that can generate a spot size of 200 μm with a power of 0.93 W and a feed of 100 mm/min was used for joining this 400 nm titanium-coated glass with polyimide. Laser energy penetrated the transparent part of polyimide imidex (PI) and was absorbed by absorbing part of titanium, so that the heat was induced directly at the interface and formed the bond line. The transmission joining configuration is shown in Fig. 1. The laser bonded joint area is 6.5 mm long. A clamping pressure of 60 psi was applied for the fabrication of the joints. We have already identified the appropriate clamping pressure for this system, which is necessary to prepare effective joint between them (Ref 4). The entire system is custom built at Fraunhofer, center for laser technology.

2.3 Bond Strength Evaluation

The laser bonded samples were tested for bond strengths using a 6-axis sub-micron tester (Ref 22). The samples were subjected to uniaxial tension after loading in the fixture of the testing machine. The average shear stress is calculated from Eq 1.

$$\text{Average shear stress} = \text{average load}/(\text{bond width} \times \text{bond length})\ (\text{N}/\text{mm}^2) \quad \text{(Eq 1)}$$

The dimensions of the prepared titanium-coated glass/polyimide lap shear test samples are shown in Fig. 2. The polyimide sheets were made of thermoplastic polyimide Imidex (Westlake Plastic Company, Lenni, PA, USA). The thickness of PI used for this study is 0.18 mm as the vendor particularly provided this thickness. Literature shows in thin films internal tensile stress is developed during deposition process (Ref 23). Besides, residual stress is also developed during laser joining process in the bond line due to mismatch of coefficient of thermal expansion (CTE) between titanium coating, glass and polyimide imidex (PI). The thickness of the titanium coating could be varied (Ref 24) and our simulation allows us to see the effect of film thickness on residual stresses developed during laser joining process. But in this particular article we did not show those results as they are not within the scope of this study.

2.4 Microscopy

For both titanium and polyimide, the failed surfaces were examined using Olympus mode BX51 optical microscope.

2.5 SEM Analysis

The failed surfaces of tensile-tested specimens were examined using the scanning electron microscope (XL 30 SEM-FEG/EDS) at excitation voltage equal to 30 keV in the secondary electron mode. To reduce the charging effect, the samples were coated with a thin layer of gold prior to examination by SEM.

2.6 XPS Analysis

The failed surfaces of samples were also examined by using x-ray photoelectron spectroscopy (XPS) for gathering information about the chemical bond. XPS analyses were performed using a Perkin Elmer 5500 X-ray photoelectron spectrometer equipped with a monochromatic AlKα (1486.6 eV) source. Elements present on the sample surface were identified from a survey spectrum recorded over the energy range 0-1200 eV at pass energy of 117.5 eV and a resolution of 1.0 eV. High-resolution (0.05 eV) spectra were then recorded for relevant photoelectron peaks, at pass energy of 23.5 eV, to identify the chemical state of each element. For all spectra energy referenced to 284.6 eV for the binding energy (BE) of the C1s component.

3. Results and Discussion

The bond strength produced for this sputtered 400 nm Ti-coated glass/Imidex system is about 29 MPa (n = 21) with a standard deviation of 6.6 MPa. Figure 3 shows the representative optical microscopic images of titanium-coated glass surface and Imidex (PI) surface of the lap shear tested samples. The results clearly show glass failure. Although, just from the optical micrographs a precise conclusion is not yet possible.

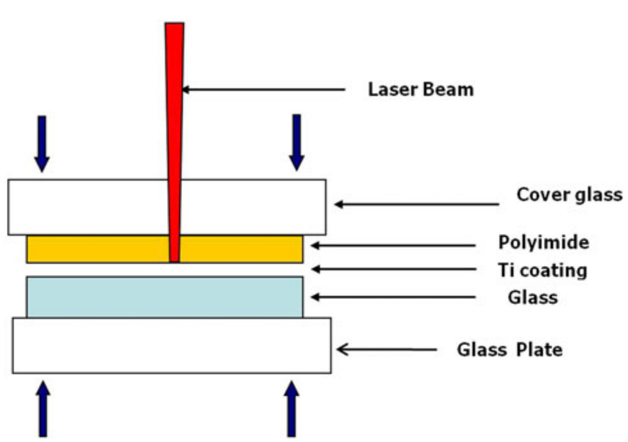

Fig. 1 Laser transmission joining of two materials using a laser beam

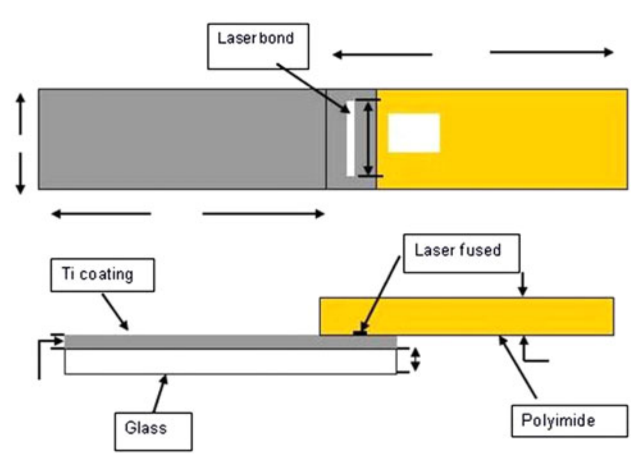

Fig. 2 Schematic of Ti-coated glass/polyimide sample (all dimensions in mm)

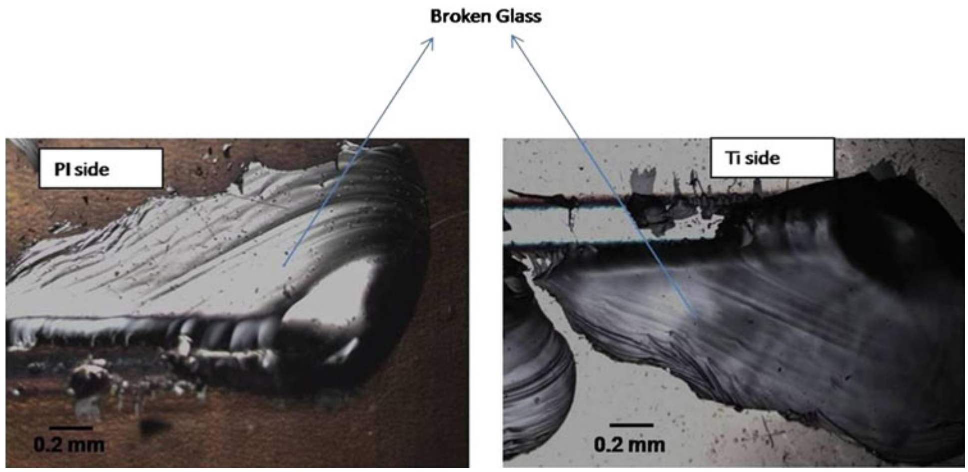

Fig. 3 Optical microscopic images of failed Ti-coated glass and polyimide surface

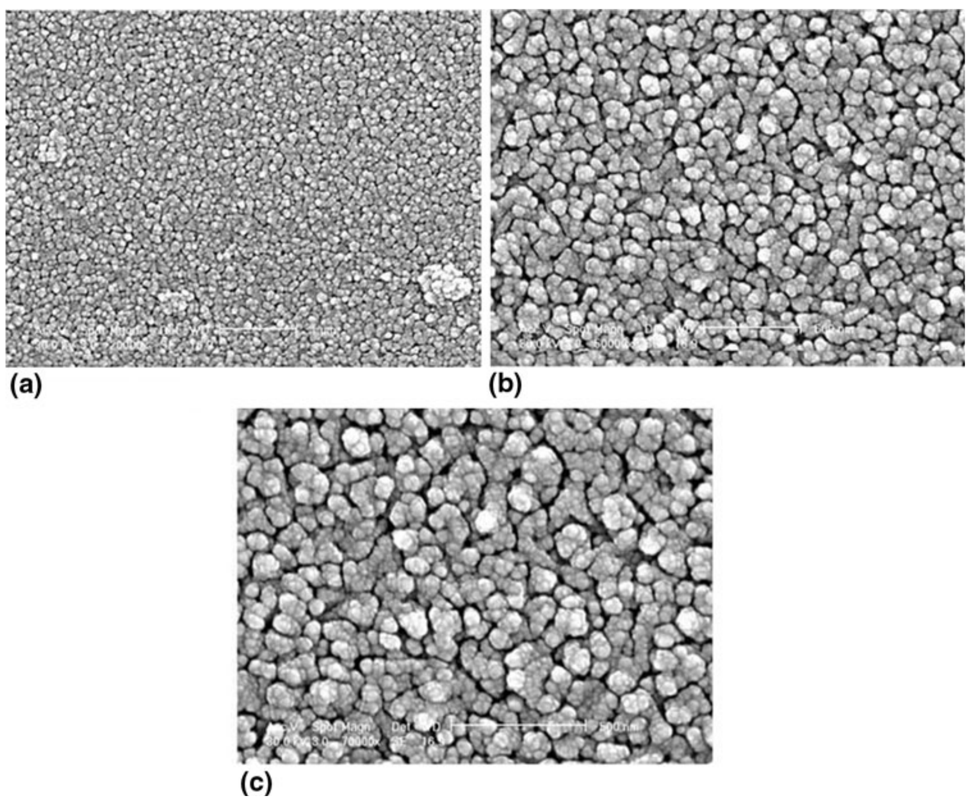

Fig. 4 SEM Images of surface morphology of sputtered Ti film (film thickness 400 nm) at different resolutions (a) 20000× (b) 50000× (c) 70000×

Figure 4 shows the SEM micrographs of 400 nm sputtered Ti-coated film on glass at different magnifications. It can be seen clearly that the films have perfect uniformity and homogeneity. Another important observation was no peeling of titanium film which means better adhesion of film to glass substrate. The average surface roughness obtained for 400 nm Ti-coated glass is 17 nm, which is 5.6 times higher when compared to thinner film of 50 nm (Ref 20). The reason for the highest surface roughness is probably due to coarser structure of the film, called grains. Roughness enhances mechanical interlocking property and ultimately bond strength increases. From SEM analysis (Fig. 5a), it was seen that a good amount of imidex was transferred to the Ti surface and also the crack initiation and propagation of the film occurred on the edge of the joint line. A portion of this polymer layer was torn up and attached to the titanium surface (Fig. 5a). This kind of phenomenon (partial torn up) indicated that cohesive failure of polymer was taking place instead of Ti-coated glass/PI

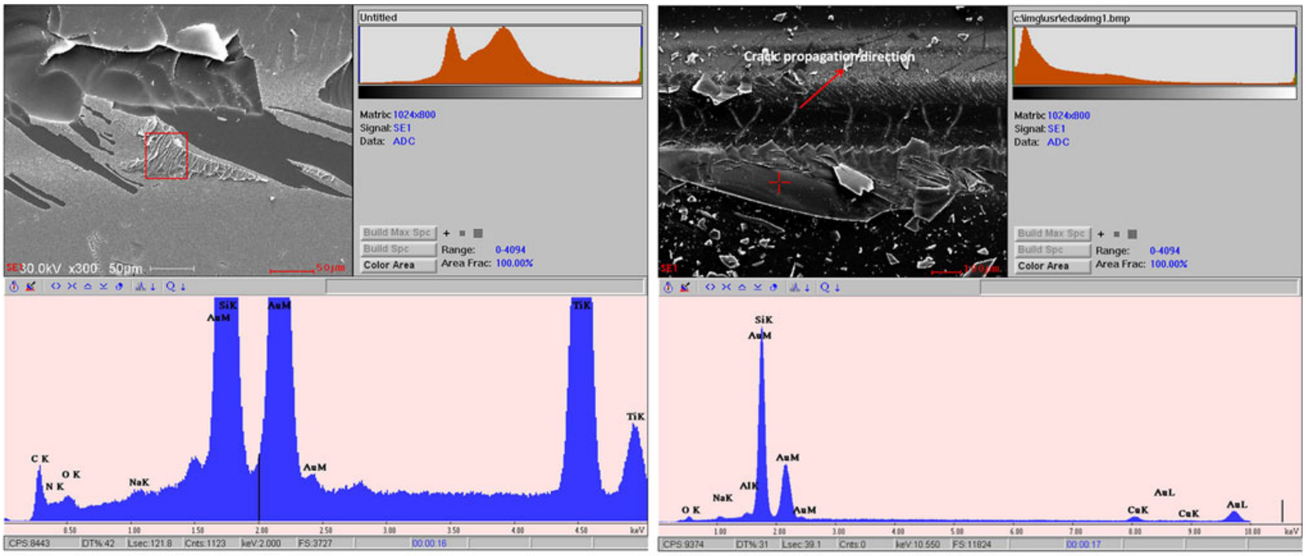

Fig. 5 (a) SEM micropraph/EDS spectrum of failed Ti surface of Ti-coated glass/polyimide sample; (a) showing presence of imidex residue and (b) showing broken glass pieces as well as crack propagation direction

interface. Presence of C and N peak from EDS spectrum of titanium surface confirms this transfer of polyimide, as the only source N is polyimide.

Figure 5(b), which is the high resolution SEM/EDS image and spectra of imidex side of the lap shear tested sample clearly showed deformation of polymer due to cohesive failure of polymer and broken pieces of glass and Ti confirms that they were transferred from the Ti surface. From the SEM/EDS analysis glass is also observed on titanium surface, this indicates interface failure of titanium/glass. Besides, the polyimide surface is showing crazes, consisting of numerous thin of stretched molecular chains which are drawn from the polymer (Ref 25-28). So, failure took place on the glass, on the Ti/glass interface and also cohesive failure on polymer; the failure pattern was mix mode.

The XPS C1s line high-resolution spectrum for failed titanium surface is shown in Fig. 6(a). Both on-joint and off-joint spectrum corresponds two intense peak, major peak at 284.6 eV due to the mixture of C-C, C-H, and C-O bond (Ref 12, 29) and the other one is from a imidex surface with contributions from carbonyl group (close to 288.4 eV). The high-resolution XPS Ti2p3 off-joint spectra (Fig. 6b) show two peaks at binding energy of 458.4 and 464.8 eV. Both the off-joint and the on-joint Ti2p3 spectra taken from the surface correspond to fully oxidized Ti, i.e., TiO_2 which has formed on the surface due to high reactivity of Ti. The Ti2p3 spectra taken on-joint area of the titanium side of the sample have lower intensity than the peak of the off-joint line. The XPS data collecting spot diameter is 2 mm which is much larger than the width of the laser joint (0.2 mm). When the polymer is peeled off from the Ti-coated glass, presence of polymer is observed on the joint spot of the titanium surface (Fig. 5a). Also Ti coating has been peeled off and broken pieces of glass are observed (Fig. 3). For the spectrum taken from the off-joint area, prominent peak corresponding to TiO_2 is observed, because 100% of the XPS signal comes from the Ti coating. For the spectrum taken from the on-joint area, part of the signal comes from the off-joint area, because the width of the laser

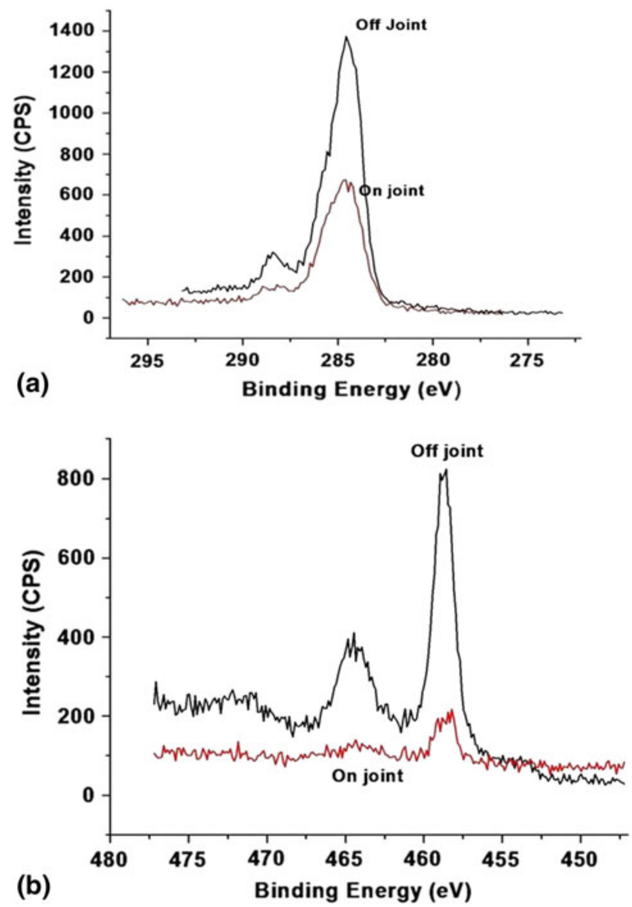

Fig. 6 XPS spectra taken for the Ti-coated glass/polyimide system on failed Ti surface (a) C1s and (b) Ti2p3 spectra

joint is less than 2 mm (which is the diameter of the collecting spot), and the rest of the signal comes from the joint area, which has pieces of polymer that covers the Ti coating and also broken glass. Presence of this polymer and glass on the joint line is responsible for less prominent peak on-joint area than the one taken from the off-joint area. Metallic Ti, TiC, and TiO (Ref 30-32) were found in the region of 452-454 eV.

4. Conclusions

Transmission laser micro-joining method has been used to join sputtered Ti-coated glass with polyimide film. The lap shear tested samples have been characterized using optical microscopy, scanning electron microscopy (SEM) along with energy dispersive spectroscopy (EDS) and x-ray photoelectron spectroscopy method. The bond strength obtained for this Ti-coated glass/polyimide sample is about 29 MPa. The results revealed that good bond strength is achieved even for a very thick film of 400 nm Ti. Sputtered Ti film thickness over 50 nm exhibits moderate surface roughness, enhanced bond strength due to mechanical interlocking property. For optical microscopy and SEM/EDS analysis of the lap shear tested samples, mixed mode failure is observed; glass failure, Ti/Polyimide interface failure and cohesive failure of polyimide. XPS investigation of the failed surface showed formation Ti-C-, Ti-O-, and TiO_2-type bonds. Those chemical bonds are essential for strong bond strength. Both bond strength and failure mode assessment results indicate consistency with previous results and also suggest that thin film of titanium with thickness ranged from over 50 to 400 nm produced strong and reliable bond.

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