Laser bonding and characterization of Kapton® FN/Ti and Teflon® FEP/Ti systems

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Abstract Kapton® FN and Teflon® FEP (fluorinated ethylene propylene) polymers are resistant to most chemical solvents, heat sealable, and have low moisture uptake, which make them attractive as packaging materials for electronics and implantable devices. Kapton® FN/Ti and Teflon® FEP/Ti microjoints were fabricated by using focused infrared laser irradiation. Laser-bonded samples were tested with a micro-testing machine under lap shear load and the bond strength was determined. The bond strength for the Kapton® FN/Ti and Teflon® FEP/Ti systems was found to be 3.32 and 8.48 N/mm², respectively. The failure mode of the mechanically tested samples was studied by using optical microscopy and scanning electron microscopy coupled with energy dispersive X-ray spectroscopy. Chemical interactions during laser bonding of Kapton® FN to titanium were studied by using X-ray photoelectron spectroscopy (XPS). The XPS results give evidence for the formation of Ti–F bonds in the interfacial region.

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Introduction

Continuous progress in science, technology, and medicine has resulted in the fabrication of various micro-electro-mechanical systems (MEMS) and BioMEMS devices. The miniaturization of the micro-systems and bio-implantable devices leads to improvement of the portability and reduction of the cost and material required. However, the small size, heat sensitivity of the MEMS components, and the need for localized joining of dissimilar materials in such systems make their packaging a challenging task. Adhesive bonding and soldering, which are conventional methods for joining of dissimilar materials, have major drawbacks such as high heat input for soldering, non-uniformity and shrinkage during curing for adhesives. These limitations can be overcome by laser-joining techniques. The advantages of the laser-joining techniques are the ability to focus the laser beam to spot sizes in the micrometer range and to precisely control the laser power in the focal spot. This control enables highly localized processing with minimum heat effect outside the joint region.

The biocompatibility, flexibility, and the ability to recover their original shape after they have been rolled or folded make the polyimides appropriate as a material for packaging of bio-implantable devices [1, 2]. A major drawback of polyimides is that they have average water uptake of 1% and water transmission rate of 1.350 g·mm/m²·day [3]. Also, some polyimides, which are not thermoplastic, such as Kapton® HN, form very weak laser bonds with other materials (unpublished results). Therefore, we have investigated the laser bonding of Kapton® FN (Kapton® HN polyimide film laminated on both sides with Teflon® fluorinated ethylene-propylene fluoropolymer) and Teflon® fluorinated ethylene-propylene (FEP) films to titanium foil. The advantage of these materials is that they have

average water uptake of less than 0.01%. Also, Kapton® FN and Teflon® FEP have water transmission rate of 0.060 and 0.178 g·mm/m²·day [3], respectively.

Previously, in work done by our group, a laser transmission technique was used to fabricate submillimeter laser bonds between titanium foil and Imidex® polyimide, which is a thermoplastic polyimide film, made from AURUM® resin, and between titanium-coated glass and Imidex® polyimide. The ranges of processing parameters that permit successful joining were identified and the bond strength was measured by lap shear mechanical tests [4]. Failure mode was investigated by using optical microscopy. The results show that the locus of failure in titanium foil/Imidex® polyimide system depends on the laser parameters at which the laser bonds were fabricated [5]. As a potential application in bio-implantable devices, the effect of the artificial cerebrospinal fluid (physiological saline with a low concentration of other salts) on the joint strength and failure mode for titanium foil/Imidex® polyimide and titanium-coated glass/Imidex® polyimide systems were studied. The joint strength decreases upon exposure to artificial cerebrospinal fluid. Scanning electron microscopy (SEM) observations of the failed samples show that the locus of failure in samples not soaked in artificial cerebrospinal fluid is deep in the polyimide polymer, while the failure of samples treated in artificial cerebrospinal fluid is of interfacial type [6, 7].

In a previous study, we have investigated Kapton® FN/Ti interfaces prepared at laser power of 4.21 W and laser speed of 100 mm/min. The results show that there is formation of Ti–F chemical compounds during laser bonding [8]. In this work, we have studied the chemical bond formation during laser bonding of Kapton® FN to titanium. The Kapton® FN/Ti sample studied was fabricated at laser power of 5.02 W and laser speed of 100 mm/min. These laser parameters, at which burning of the polymer occurred, were used intentionally for better understanding of the formation of chemical species during fabrication of the laser bonds. On fluorinated polymers such as Teflon® FEP, Teflon® PFA, FLARE™ 1.0, and PTEF, titanium deposition defluorinates the surface of the polymer and leads to the formation of titanium fluoride and titanium carbide species [9–12]. These results provide insight into the chemical bond formation during titanium deposition on the polymer surface under clean, high-vacuum conditions.

In this study, laser joints for Kapton® FN/Ti and Teflon® FEP/Ti systems were fabricated under ambient conditions. Bond strength of the laser bonds was measured and failure mode was studied by using optical microscopy and SEM coupled with energy dispersive spectroscopy (EDS). The chemical bond formation during laser bonding of Kapton® FN to titanium foil was studied by using X-ray photoelectron spectroscopy (XPS). Since the laser bonding process has been conducted in ambient conditions, the XPS results will provide insight into chemical bond formation between the FEP layer of the Kapton® FN composite and the native TiO_2 layer of the titanium foil.

Sample preparation and characterization methods

Kapton® 500FN131 0.127-mm-thick composite sheet (DuPont) (which was composed of a 0.0762-mm-thick Kapton® HN core, laminated on both sides with 0.0254-mm-thick Teflon® fluorinated ethylene-propylene (FEP) fluoropolymer) and Teflon® FEP (DuPont) sheet with thickness of 0.127 mm were laser-joined to 0.05 mm Ti foil (99.6% purity, Goodfellow Corp.) by using continuous radiation from a Yb-doped fiber laser operating at $\lambda = 1.1$ µm. The so-called transmission joining procedure [4] was used, and a schematic representation of the sample mounting and processing is shown in Fig. 1. In this procedure, the system to be joined consists of a transparent (polymer) and an absorbing (titanium foil) part at the wavelength of the laser. The laser energy passes through the polymer and is absorbed by the titanium foil, thus inducing the heat directly at the interface. The polymer part melts, which allows the formation of bond with the titanium foil. The clamping pressure, at which the samples are held during the bonding, is important to the formation of the laser micro-joints. A clamping pressure of 414 kPa was used for the fabrication of the samples studied in this work. A laser beam with total power of 4.21 and 5.50 W for the Kapton® FN/Ti and Teflon® FEP/Ti systems, respectively, was focused on the Ti surface to a spot with a diameter of 200 µm. A laser speed of 100 mm/min was used to fabricate the Kapton® FN/Ti and Teflon® FEP/Ti laser joints. The optical transmission of the polymers we used in this study was characterized by spectrophotometry and found to be 92% for the Kapton® FN and 94% for the Teflon® FEP polymer at $\lambda = 1.1$ µm.
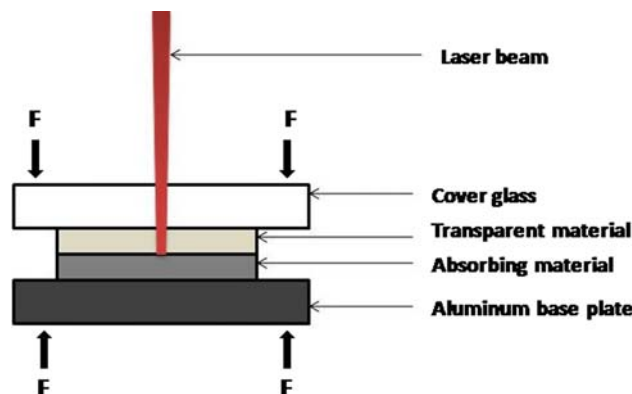
Fig. 1 Schematic diagram of transmission joining of two materials using a focused laser beam
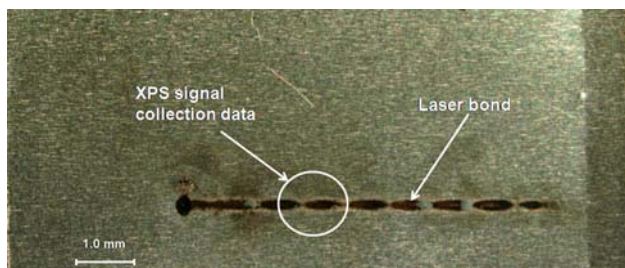
Fig. 2 An optical micrograph of the titanium side of a Kapton® FN/Ti sample used for XPS study

Fig. 3 Mechanical test sample sizes and configuration

The laser fabricated joint samples were tested for mechanical lap shear strength by using a 6-axis general-purpose micromechanical/thermal testing instrument [13]. The machine could be operated both in displacements and load controls with six degrees of freedom, i.e., three orthogonal translations and three rotations. The displacement resolution of each closed-loop, DC-motor-drive stage is maintained at 0.1 mm in translations. A capacitance gage or a linear encoder is used to measure the actual displacement of a test sample.

Optical micrographs of the polymer and titanium sides of pull-tested Kapton® FN/Ti and Teflon® FEP/Ti samples were obtained by using Nikon Optiphot System.
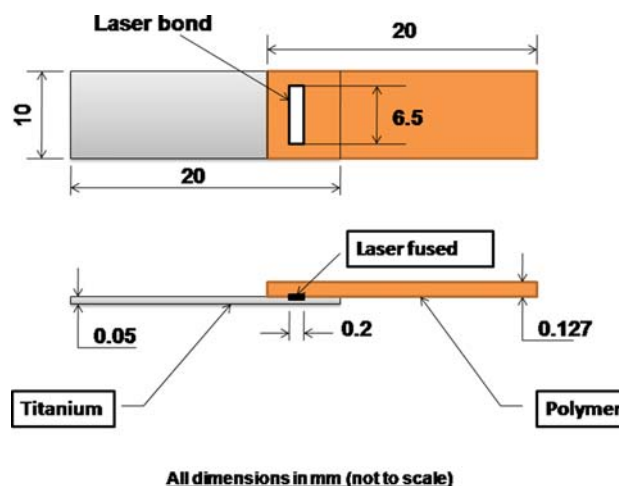
SEM images and EDS spectra of the polymer and titanium sides of pull-tested Kapton® FN/Ti and Teflon® FEP/Ti samples were obtained in Phillips XL 30 SEM-FEG/EDS and EDAX system (EDAX, Inc.). To avoid charging effects, the polymer sides of the samples were sputter-coated with a thin Au layer before taking the SEM images.

In order to study the chemical bond formation during the process of laser joining, a Kapton® FN/Ti sample was prepared at laser power of 5.02 W and laser speed of 100 mm/min. The sample was separated into its polymer and Ti parts by peeling in air and the titanium side was loaded immediately in the UHV analysis chamber of a Perkin-Elmer model 5500 XPS spectrometer with a monochromatic Al Kα X-ray source. An optical micrograph of the Ti side of the sample is shown in Fig. 2. The base vacuum pressure was 3.1×10^{-9} Torr, and an Ar-ion beam was used to sputter the sample surfaces in order to obtain spectra from various depths below the surface. A beam voltage of 4 kV and raster size of 4×4 mm was used during sputtering.

Results and discussion

Bond strength measurements and failure characterization

The mechanical test sample configuration and sizes are shown in Fig. 3. During testing, the samples were subjected to uniaxial tension in a direction perpendicular to the laser bond. Data for the failure loads were collected. The bond strength for all samples was calculated by normalizing the failure loads by the joint area as measured by optical microscopy. The bond strength results for Kapton® FN/Ti and Teflon® FEP/Ti systems are summarized in Table 1. Even though the bonding for the Kapton® FN/Ti system actually occurs between the thin Teflon® FEP layer and the Ti, the bond strength of the Kapton® FN/Ti laser joints is 2.5 times less than the bond strength of the bulk Teflon® FEP/Ti laser joints. A possible explanation of this is the fact that the Kapton® FN material is a composite consisting of three layers. In this case, the thin Teflon® FEP layer is constrained between the Ti and thicker, higher modulus Kapton layer. Thus, upon shear bond strength testing, the ratio between the normal and shear stresses at the Ti-FEP interface for the Kapton® FN/Ti system is different from the normal/shear stresses ratio for the bulk Teflon® FEP/Ti system. This leads to difference in the force field, formed during the shear strength testing, for the two systems. These mechanical differences may account for the observed difference in strength, and this effect will be investigated further.

Previously we have investigated the Ti coated glass/Imidex® polyimide system. The highest bond strength for this system was found to be 24 N/mm² [6]. In another work done in our laboratory, we have found that the highest bond strength for the poly(vinylidene fluoride)/Ti system is 6.7 N/mm² [14].

Figures 4 and 5 show the optical micrographs of the titanium and polymer side of lap shear tested Kapton® FN/Ti and Teflon® FEP/Ti samples, respectively. Inspection of the titanium side of failed Kapton® FN/Ti joint under an optical microscope (Fig. 4a) showed that there is a small amount of polymer residue on the titanium side of the Kapton® FN/Ti sample. This means that the locus of failure

Table 1 Bond strength results

System tested	Number of samples tested	Laser power (W)	Laser speed (mm/min)	Bond strength (N/mm^2)
Kapton® FN/Ti	6	4.21	100	3.32 ± 0.16
Teflon® FEP/Ti	6	5.50	100	8.48 ± 0.67

Fig. 4 Optical micrographs of lap shear tested Kapton® FN/Ti sample: **a** titanium side of the sample; **b** Kapton® FN side of the sample

KaptonFN residue on failed Ti surface
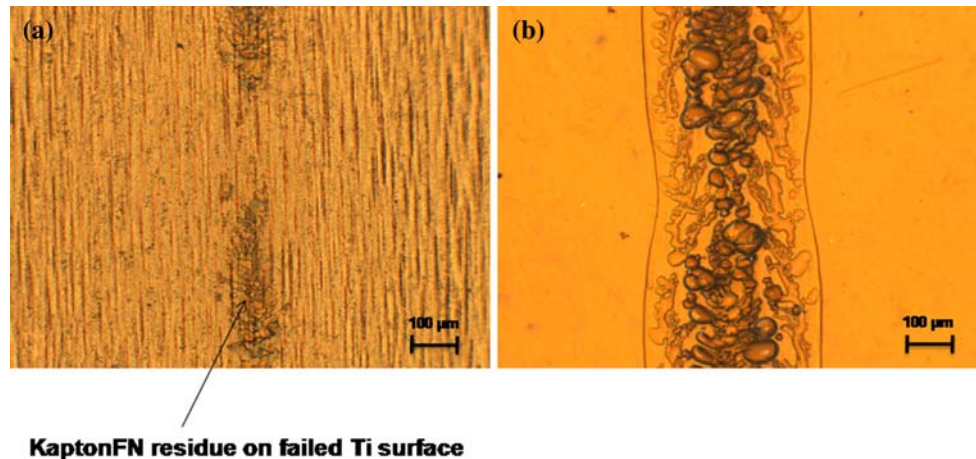
Fig. 5 Optical micrographs of lap shear tested Teflon® FEP/Ti sample: **a** titanium side of the sample; **b** Teflon® FEP side of the sample


Teflon® FEP residue on failed Ti surface
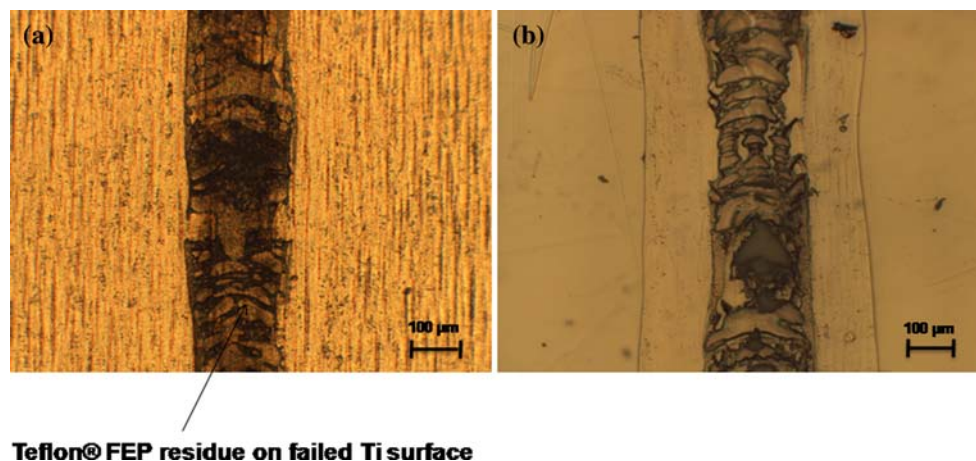
is close to or at the Kapton® FN/Ti interface (interface failure). Examination of the titanium side of lap shear tested Teflon® FEP/Ti sample (Fig. 5a) showed that the failure of this system occurs away from the interface, deep in the Teflon® FEP (cohesive failure), which results in large amount of Teflon® FEP polymer residue on the failed titanium surface.

In order to establish in which layer of the Kapton® FN composite is the locus of failure, the chemical composition of the polymer residue found on the titanium side and the bond area of the polymer side of a failed Kapton® FN/Ti sample were studied by using SEM coupled with EDS technique. The same technique was used to study the titanium and polymer side of a lap shear tested Teflon® FEP/Ti sample.
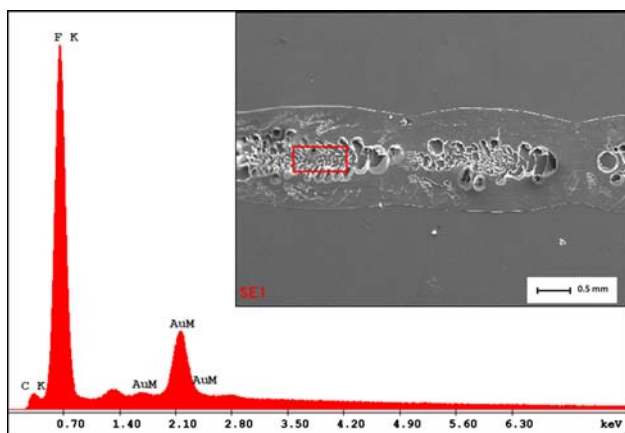
In Figs. 6 and 7, the SEM images and the EDS spectra taken from the joint area of the titanium and Kapton® FN side of a lap shear tested Kapton® FN/Ti sample are shown, respectively. After lap shear testing, polymer residue was found on the titanium side of the sample. In order to investigate the failure of the sample, the EDS spectrum was obtained from this polymer residue (Fig. 6). The EDS analysis showed only the presence of F and Ti. The EDS spectrum, taken from the polymer side of the sample (Fig. 7), showed the presence of F and C. The only source of fluorine can be the Teflon® FEP layer of the Kapton® FN polymer. On the other hand, the Kapton® HN core of the Kapton® FN composite contains N and O. The fact that no N and O were found on the failed titanium and Kapton® FN side of the sample and no Ti was found on the polymer
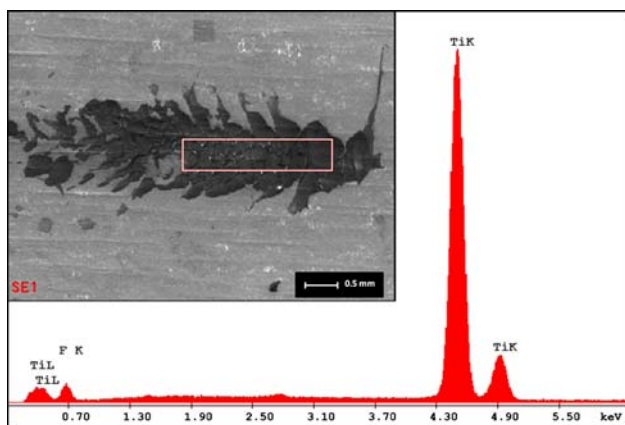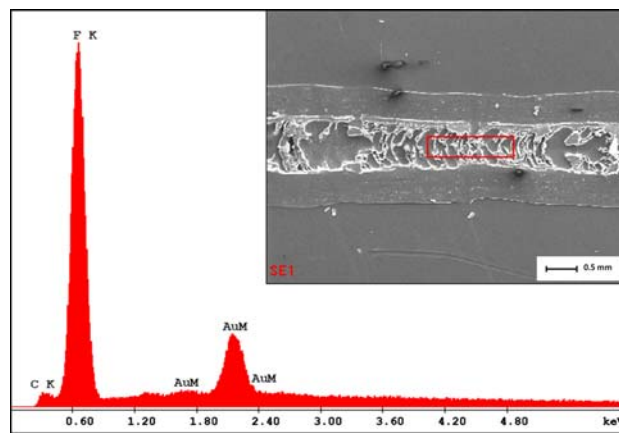
Fig. 6 SEM image and EDS spectrum taken from the titanium side of a lap shear tested Kapton® FN/Ti sample. The *rectangle* indicates the area analyzed in the EDS spectrum
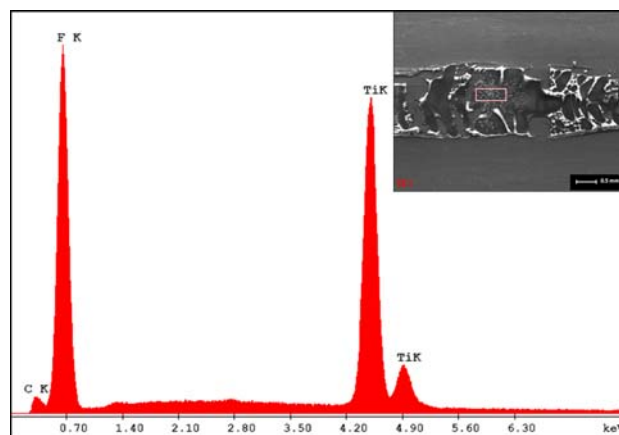
Fig. 8 SEM image and EDS spectrum taken from the titanium side of a lap shear tested Teflon® FEP/Ti sample. The *rectangle* indicates the area analyzed in the EDS spectrum

Fig. 7 SEM image and EDS spectrum taken from the Kapton® FN side of a lap shear tested Kapton® FN/Ti sample. The *rectangle* indicates the area analyzed in the EDS spectrum

Fig. 9 SEM image and EDS spectrum taken from the Teflon® FEP side of a lap shear tested Teflon® FEP/Ti sample. The *rectangle* indicates the area analyzed in the EDS spectrum
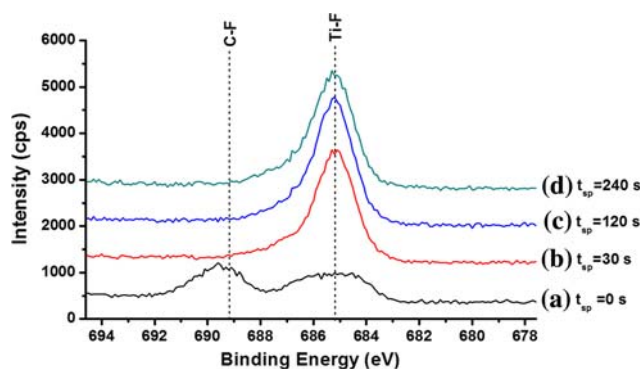
side of the sample suggests that the locus of failure for the Kapton® FN/Ti system is inside the Teflon® FEP layer close to the interface.

In Figs. 8 and 9, the SEM images and the EDS spectra taken from the joint area of the titanium and Teflon® FEP side of a lap shear tested Teflon® FEP/Ti sample are shown, respectively. The EDS spectra taken from the titanium side (Fig. 8) of the sample showed the presence of C and F. Their source is the Teflon® FEP polymer. This is in agreement of the fact that the joint area of the Ti side of the sample was covered with continuous layer of polymer residue. The EDS spectra taken from the Teflon® FEP side (Fig. 9) of the sample showed the presence of C and F. No presence of titanium was detected on the polymer side of the failed sample. These results suggest that the failure for the Teflon® FEP/Ti system occurs deep inside the Teflon® FEP polymer away from the Teflon® FEP/Ti interface.

X-ray photoelectron spectroscopy results

A Kapton® FN/Ti sample prepared at laser power of 5.02 W and laser speed of 100 mm/min was studied. Both low-resolution (survey) and high-resolution (multiplex) XPS spectra were collected. The width of the joint was measured to be 200 μm. Since this width was less than the width of the area analyzed by the XPS instrument, all spectra that were collected contained an ∼10% contribution from the actual bond area and ∼90% contribution from the non-laser-treated surface of the sample. The binding energy scales were adjusted using the C1s line at 284.6 eV.

The spectra taken from the Ti side of the sample show the presence of fluorine on the titanium surface. No nitrogen was found on the titanium side of the sample. The only source of fluorine could be the FEP layer of the Kapton® FN and the source of nitrogen would be the Kapton® HN core
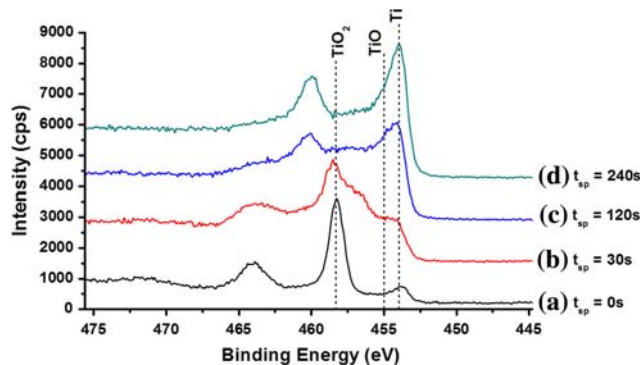
Fig. 10 F1s XPS spectra taken from the Ti side of the sample after sputtering for the indicated times (a–d)

Fig. 12 Ti2p XPS spectra taken from the Ti side of the sample after sputtering for the indicated times (a–d)

polyimide of the Kapton® FN composite. These results suggest that the locus of failure is inside the FEP layer, close to the interface.
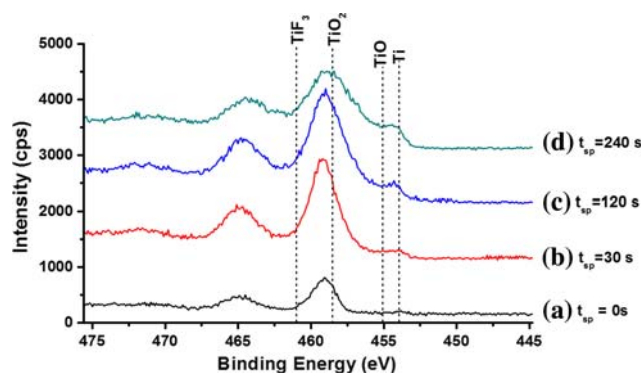
In Fig. 10, F1s spectra taken from the Ti side of the sample are shown. The spectrum taken from the surface consists of two peaks at 689 and 685 eV. The peak at 689 eV corresponds to the fluorine bound to carbon from the polymer chain [15]. The second peak at around 685 eV is in agreement with the literature data for fluorine bound to titanium [12, 16]. Upon sputtering, the peak, which corresponds to the fluorine from the FEP layer of the Kapton® FN, disappears and the spectra indicate only the presence of fluorine bonded to titanium.

Ti2p spectra taken from a non-treated Ti foil and from the Ti side of the sample are shown in Figs. 11 and 12, respectively. The vertical dashed lines indicate the positions of the Ti2p$_{3/2}$ line of TiF$_3$, Ti, TiO$_2$ and TiO [12, 17, 18].

The spectrum taken from the surface of the non-treated titanium foil consists of TiO$_2$ and metallic Ti with a Ti2p$_{3/2}$ binding energy of 458.5 and 454 eV, respectively. The Ti2p$_{3/2}$ peak, corresponding to TiO$_2$, in the spectrum taken from the titanium side of the sample is shifted to higher binding energy toward the TiF$_3$ line, indicating the

presence of TiO$_2$ and Ti–F species on the titanium surface. After sputtering for 30 s, the spectrum taken from the non-laser-bonded titanium foil consists of TiO$_2$ and metallic Ti. The shoulder, which appears at 457 eV, can be attributed to ion beam reduction of the surface. Upon further sputtering, the spectra consist predominantly of metallic titanium and TiO contribution (Fig. 11c, d). The spectrum taken from the Ti side of the laser bonded sample after sputtering for 30 s consists of mixture of TiO$_2$ and Ti–F species as well as contribution from metallic titanium. The spectra taken after sputtering for 120 and 240 s (Fig. 12c, d) consist of a state with peaks at 459 and 465 eV. This component taken along with the evolution of the fluorine spectra can be attributed to the presence of mixture of Ti–O and Ti–F bond containing compound [12, 19]. Also, the peak at 454 eV, which corresponds to metallic Ti, gains intensity in the spectra taken from the titanium side of the sample after sputtering for 120 and 240 s.

Conclusions

Laser bonds for the Kapton® FN/Ti and Teflon® FEP/Ti systems were fabricated. The lap shear strength of the laser bonds was measured. The lap sheer strength of Teflon® FEP/Ti laser bonds was measured to be 2.5 times higher than the strength of Kapton® FN/Ti system.

Failed samples were studied by using optical microscopy and scanning electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS). The results from the EDS analysis of the Kapton® FN/Ti system showed that no N was found on the laser joint area of the failed titanium and polymer part of the sample and no Ti was found on the polymer side of the sample. Also, no N was detected on the Ti side of the sample by using XPS. Since the source of N would be the Kapton® HN core of the Kapton® FN composite, these results suggest that the failure for Kapton® FN/Ti system occurs in the Teflon®

Fig. 11 Ti2p XPS spectra taken from a non-treated Ti foil after sputtering for the indicated times (a–d)

FEP layer close to or at the interface (interfacial type of failure). The EDS analysis of the Ti part of a failed Teflon® FEP/Ti sample showed the presence of C and F. No Ti was found on the polymer side of the sample. These results along with the observation, obtained from optical microscopy and SEM, of a continuous layer of polymer residue on the titanium side of the failed sample suggest that the failure of the Teflon® FEP/Ti system is a cohesive type of failure in the polymer.

Chemical bond formation during laser bonding of Kapton® FN to titanium was studied by examination of the Kapton® FN/Ti interface with XPS. The results give evidence for the chemical bond formation between F from the Teflon® FEP layer of the Kapton® FN composite and the titanium foil.

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