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ATMOSPHERIC PRESSURE GLOW DISCHARGE (APGD) PLASMA GENERATION AND SURFACE MODIFICATION OF ALUMINUM AND SILICON Si (100)

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Atmospheric pressure glow discharge was generated by AC 50 Hz power source. Argon used as the main plasma gas was electrically excited to give the APGD plasma inside a stainless steel chamber. The plasma was characterized by optical emission spectroscopy in the range 350-1050 nm with 1.5 nm resolution. Availability of oxygen in the emission spectrum was confirmed by the presence of atomic oxygen spectral lines at 777.2, 844.6 and 926.6 nm. The oxygen activated well characterized plasma was exposed to Si (100) and Al for 11-60 min. Incorporation of oxygen in Si and Al surfaces was verified by the emergence of a strong peak of oxygen in the energy dispersive x-rays spectrum of the substrate. Thus oxidation of Si and Al to give SiO_2 and Al_2O_3 was successfully achieved. It was critically noted that presence of nitrogen is a major part of atmosphere, but nitrogen was neither excited nor incorporated in the Si and Al substrates. So no evidence of formation of silicon nitride or aluminum nitride was found. All measurements were taken at normal temperature and pressure.

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Keywords: Plasma generation, FTIR, Surface modification, Oxidation

1. Introduction

Over the past 40 years, plasma has become a very useful method for surface modification and deposition of various materials. An ionized medium consisting of electrons, ions, neutral species and photons, is called plasma [1,2]. According to this definition, the term plasma covers a wide range of phenomena. Surface property changes such as improved mechanical, chemical, optical and other properties using surface modifications play a significant role in materials applications in the areas such as fusion research, nano-science and technology, nuclear technology, space technology, semiconductor technology, biomedical science, engineering & manufacturing etc [3-5]. However, deeper understanding of the surface modifications at the nano-scale and atomistic level is required for the controlled processing of material surfaces in specified applications.

While all the four states liquid, solid, gas and plasma affect the surface in different ways, the vacuum is helpful in either processing or studying the processed surfaces in controlled conditions. Many phenomena, processes and properties such as thin film deposition, surface modifications, surface erosion under various interactions, corrosion, catalysis etc. are surface specific and require specialized techniques to understand them completely [6,7]. Plasma sputtering is an important part in the plasma processing to remove surface contaminants and any other layer before the process starts [8-11]. However, sputtering can lead to the compositional and/or

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morphological and topographical changes which are important in some applications. SEM study gives the details of such surface modifications. During plasma processing such as plasma ion implantation and plasma nitriding, sputtering of the material takes place [12-14]. Sputtering is also used as the first step of in-situ cleaning of the sample surface in these processes. It is known that for the multi-component system like any alloy, there is a possibility of preferential sputtering of some component leading to the compositional changes in the near surface region. Sputtering can be used as a complete processing technique to improve the surface properties such as adhesion [15]. The low temperature cold plasma has the special benefits for processing [16]. All plasma enhanced chemical processing are the result of chemical reactions, the main difference from the various processes is the state of the final product. In the deposition of the films the product will non volatile and deposit on the substrate. In some surface modification applications, such as oxidation [17] and nitriding [18], the gas is dissociate in the plasma, radicals are formed that react with the substrate surface, and a non volatile product that deposit on the surface and changes the characteristics of the substrate surface. For cleaning, etching, or ashing the products of the reactions between plasma species and substrate have to be volatile to remove material from the exposed surface [19-21]. The oxygen plasma used for the growth of the oxide layer on metal or semiconductor substrate for the surface modifications on the behalf of cold plasma termed as Oxidation. For the oxidation of surface of a semiconductor or conductor an inert gas such as argon is used with the oxygen gas for the formation of plasma. In oxidation when the bombardment of ions is used, the growth rate of oxide and the removal rate due to sputtering will be in equilibrium. By adjusting the parameters of the plasma, such as pressure, power, ratio of oxygen / argon, the oxide layer thickness can be controlled [22].In this study the plasma generation at atmospheric pressure with AC 50 Hz power source was performed and oxidation of Silicon (Si) and Aluminum (Al) were discussed by plasma implantation.

2. Experimental work

In the chamber first the vacuum was created. The argon gas was filled with the help of the flow meter, this gas is non reactive called the noble gas. When the chamber pressure was reached to 990 mbar the supply of the gas was stopped. The temperature of the laboratory was measured 26 $^{\circ}$C at that time. The two electrodes of aluminum in the plane geometry were used. The spacing between the electrodes was 2.0 mm. The material silicon Si (100) whose surface modification was required cut in a dimension of 10 x 8 mm^2 in the plane shape. The plane silicon piece has two surfaces one was polished and the other was rough. The silicon rough surface was placed on the aluminum electrode and, the polished surface was at the upper side in between the electrodes. The electrodes wires were connected to the output of the step up transformer. The input of the step up transformer was connected to the AC main and the voltage is gradually increased at a certain high voltage the plasma was generated between the two electrodes. The transformer which was used gives the voltage about 15000 V (15kV), at the 220V of primary input. Its mean when the input voltage of the transformer was increases by 1 V the output of the transformer was increased by 68 V approximately. The Si (100) plane was placed in front and the voltage was increased gradually and at a certain voltage, 40 volts at the input of the transformer or 2720 volts (40x68 V) at the output of the transformer, which was the voltage between the electrodes, the plasma was generated. The transformer input voltage was set at 50 V at that time the transformer output voltage which was the voltage between the electrodes was 3400 V (50 x 68). The current flowing at that time was 2.42 mA. The generated argon plasma was fall for 11 minutes on the silicon surface. After the plasma fall the silicon sample was removed from the chamber. Similarly other three samples of silicon and four samples of aluminum were prepared for surface modification purpose, for different time and having the different dimensions, but in the same argon plasma at the same pressure and AC (50 Hz) source. The details of these samples preparation are shown in the Table 1. The plasma was characterized by a CCD type spectrophotometer with 1.5 nm resolution. Optical emission spectroscopy (OES) was taken to measure the electron temperature and electron density of electrons present in the chamber. The Scanning electron microscopy (SEM) model S-3000H (Hitachi) was used for the investigation of surface morphology and Energy

Dispersive X-ray spectroscopy (EDX) analysis, which was equipped with SEM was performed to confirm the elemental composition. Fourier transformed infrared (FTIR) spectroscopy was also performed for bonding information among different elements.

Table 1. Silicon Si (100) and Aluminum (Al) samples preparation detail.

Substrate	Dimension of Substrate (mm^2)	Exposure Time (min)	Electrode Voltage (volt)	Current (mA)	Inter Electrode Spacing (mm)	Pressure (mbar)
Si(100)	10 x 8	11	3400	2.42	2.0	990
Si(100)	7 x 5	25	4760	3.95	2.0	990
Si(100)	8 x 7	40	3400	2.75	2.0	990
Si(100)	6 x 6	60	3400	2.77	2.0	990
Al	10 x 10	11	1730	1.38	2.5	990
Al	10 x 10	25	1840	1.42	2.5	990
Al	10 x 10	40	2040	1.62	2.5	990
Al	10 x10	60	2720	1.75	2.5	990

3. Results and discussions

3.1 Optical emission spectroscopy

The OES spectrum is also taken for the argon plasma generated in the chamber shown in the Fig. 1.The emission spectrum of plasma gives the information about the electron temperature and electron density [23]. There are many methods in OES for finding the electron temperature and electron density. The most convenient and popular techniques of determining electron temperature is the two-line-emission ratio method, which yields the electronic excitation temperature (T_{exc}) that can then be equated to the electron temperature(T_e). The two lines which have same lower energy level have been selected for the determination of the excitation temperature. The formula is used to determine the electron temperature in intensity ratio method is,

$$T_e = \frac{(E_2 - E_1)/K}{\ln(\frac{A_1 g_1 I_2 \lambda_2}{A_2 g_2 I_1 \lambda_1})}, \quad (1)$$

Where A_1 is transition probability of 1st line, A_2 is transition probability of 2nd line, g_1 is statistical weight of 1st line, g_2 is statistical weight of 2nd line, I_1 is intensity of 1st line, I_2 is Intensity of 2nd line, λ_1 is wavelength of 1st line, λ_2 is wavelength of 2nd line, E_1 is upper level energy of 1st line, E_2 is upper level energy of 2nd line, and K is Boltzman constant.

Electron number density was calculated by full width at half maximum for various emission lines by the relation given below called stark broadening method [24].

$$n_e = 3.71 \times 10^{14} (\Delta\lambda_{1/2})^{1.431}, \quad (2)$$

The electron temperature is calculated to be 0.56 eV (6500 K) and electron density to be 1.7 x 10^{16} electrons par cm^3 respectively. The surface modification of the materials such as silicon and aluminum was discussed by argon plasma. First surface modification of silicon and after that of aluminum is discussed. Since modification means the changes that occur at the surface of the material. This change may be any type, such as the crystallinity of the material at the surface may destroyed, the surface may be damaged and the chemical reaction of any other element may be taking place [25].

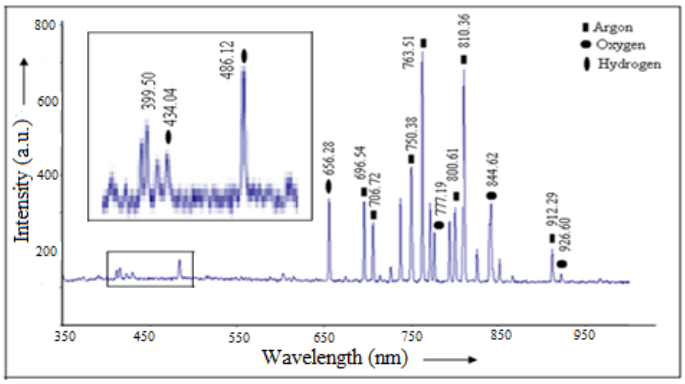

Fig. 1 Optical emission spectrum of argon plasma.

3.2 Surface Modification of Silicon Si (100)

3.2.1 SEM/EDX Analysis

SEM micrograph and EDX graph of the silicon Si (100) were taken before exposed of argon plasma, which were not shown here. It is clear from the micrographs the Si (100) has the plane surface and EDX graph confirms that, there is no other element on the surface of the Si (100). For the investigation of the surface of the silicon the SEM microscopy images ware taken, for the silicon samples which were prepared for 11, 25, 40 and 60 minutes as shown in Fig. 2 (a), (b), (c), and (d) respectively. The magnification was x 40 for samples shown in Fig. 2 (a) and (b), x 100 for Fig. 2 (c) and x 200 for Fig. 2 (d). The SEM image of the Si (100) shows that the plane polished surface of the silicon was changed (modified). The surface of the silicon shows that the argon ions which were heavy and non reactive strikes the surface and sputter (eject) the silicon atoms from the surface [26]. From Fig. 2 (a) and (b) it is observed that when the time of plasma exposed increases the more changing occur at the surface. If the plasma exposure time was further increase, there is a significant changed occur at the surface. In Fig. 2 (c) and (d) it is clearly observed that there is a layer of other materials are formed on the surface of the silicon, because the surface of the silicon become invisible. SEM microscopy just shows the information of the physical appearance of the material. Physically we can see surface is changed or not, but something new is formed or any compound is formed, that is not investigate from the SEM microscopy. Because first the chamber was evacuated from air and then the argon gas was filled and since argon is a non reactive gas, so there was a very less chance the silicon make compound with other element, because there is no element except argon and aluminum (electrodes) present in the chamber in ideal case. But SEM images show that something new is formed on the surface. So for the investigation of the new materials on the surface EDX analysis was performed.

In the SEM an integrated unit of the EDX was also present so an EDX measurement was also taken for elemental composition. The EDX graphs were taken for samples which were prepared for 11, 25, 40 and 60 min the representative graph is shown in Fig. 3. In the EDX graph three elements are shown oxygen, silicon and aluminum. The silicon is present in the EDX analysis, because the substrate was of silicon. The aluminum is present in the graph, because the electrodes were used of aluminum. It is due to some atoms of the aluminum may sputter from the electrodes, and stick or deposited on the silicon surface. Oxygen is also present in the graph. The oxygen is present in the air, when the chamber was evacuated some of air (oxygen) molecules may enter into the chamber. These molecules of the oxygen will change into the reactive species in the argon plasma, and these species reacts with silicon surface, and a bond was formed between the silicon and oxygen. In the air a large amount of nitrogen is also present i.e. 78%. But the nitrogen makes a triple bond (N≡N) which will require a large amount of energy (9.8 eV). On the other hand, the oxygen makes double bond (O) requires less energy (5.1 eV) [27]. So nitrogen will not convert into ionic or reactive species. That is why the reaction between silicon and oxygen dominants than silicon and nitrogen. Usually the oxygen makes a negative ion (O^-) in the plasma and nitrogen does not make the negative ion so there is a very less chance for nitrogen to react

with the silicon surface. But it is also believed that the oxygen atom is more responsible for the oxidation of the silicon than the oxygen negative ion.

So from the results and the above discussion the SEM micrographs show that the layer of the new material is formed on the silicon substrate and the EDX analysis verify that the oxygen layer is formed on the silicon substrate. Optical emission spectroscopy (OES) spectrum was also taken for the argon plasma generated in the chamber is shown in Fig. 1. In the spectrum the most of the peaks are present for argon at the wavelengths (696.54, 706.72, 750.38, 763.51, 800.61, 810.36, and 912.29 nm), which are shown by rectangular marks in the spectrum above the wavelengths peaks. Some oxygen peaks are also present in the spectrum at wavelengths (777.19, 844.62, 926.60 nm) [28], which are shown by the circular marks above the wavelengths peaks. The wavelength peaks for argon (Ar) and oxygen (O), which are shown in the spectrum verified by the National Institute of Standards and Technology (NIST) data. From the above information there are more peaks for argon and some peaks for oxygen, confirms that the oxide layer is formed on the silicon.

For the films generation the inert gases (argon), which is also an atomic gas is used for the plasma generation with molecular oxygen (O_2). Because plasma can be easily generate in the atomic gases than the molecular gases. In air a large amount of nitrogen is present, but the OES spectrum shows no peak for nitrogen. This shows that the nitrogen will not change into excited atoms or in reactive species that is why it will not react with silicon in this case.

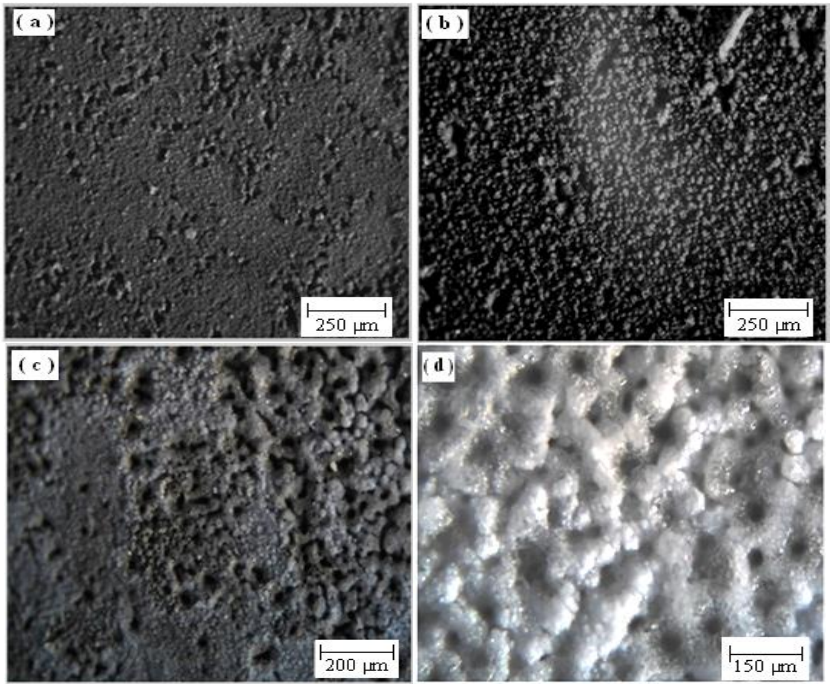

Fig.2 SEM micrographs of Si (100) after exposure to argon plasma for (a) 11 (b) 25 (c) 40 and (d) 60 min.

Semi-quantitative analysis were performed using an energy dispersive spectrometer (EDS) attached with SEM, also gives the composition of the material in terms of weight % [29]. Since four samples of the silicon were prepared after plasma exposed for 11, 25, 40 and 60 min. The weight % of the oxygen is 17.03, 27.51, 28.68, and 29.51 and for silicon is 6.46, 5.14, 4.36, and 3.21 for the samples on which the plasma was exposed for 11, 25, 40 and 60 min respectively. It is obvious that the weight % of the oxygen is increased 17.03, 27.51, 28.68 and then 29.51, when the plasma exposure time was increased [30]. It means when the time for plasma exposed was increased then the oxygen layer or oxidation of the silicon increases [31]. The other thing which is also observed that, the weight % of the silicon decreases with increase of time of plasma implantation. The reason of the decrease in the weight % of the silicon is that, when the plasma

expose time increases the oxidation increases means the layer of oxygen atoms on the silicon substrate increases. Since in the EDX principle the high energy electrons strikes the sample and the sample atoms become excited or ionized due to this excitation and ionization the electrons transition in the atoms of the sample from higher energy levels to lower energy levels and the characteristics rays (photons) are emitted of the specific energy. EDS plots a graph and senses the elements present in the sample. When the oxidation is increased on the silicon substrate, then that high energy electrons just strike on surface atoms that will be of oxygen. Silicon surface is covered by the oxygen atoms due to which EDS senses the oxygen in more weight % than that of silicon. The conformation in the increase of oxidation is physically visualized by the SEM images.

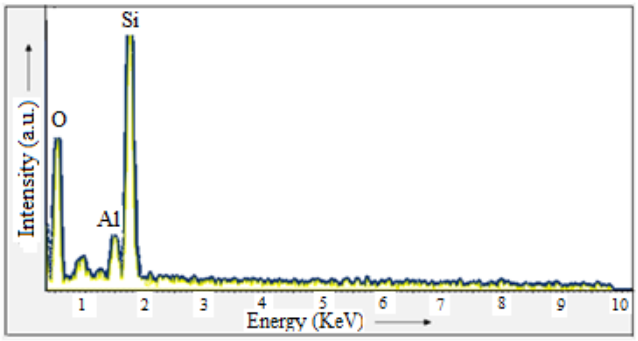

Fig. 3 EDX graph of Si (100) after exposure to argon plasma for 40 min.

3.2.2 FTIR Analysis

For the conformation of the silicon oxygen bonding and aluminum oxygen bonding FTIR analysis of the same Si (100) samples were performed. The samples which were exposed for 11 and 25 minutes by plasma are shown in Fig. 4 (a) and (b), and which were exposed for 40 and 60 minutes shown in Fig. 4 (c) and (d) respectively. The characteristics bands at 809.60 (Fig. 4 (a)), 818.51 (Fig. 4 (c)) and 805.29 cm^{-1} (Fig. 4 (d)) shows the Si-O bending. The peaks at 1091.76 (Fig. 4 (a)), 1105.20 (Fig. 4 (c)) and 1112.35 cm^{-1} (Fig. 4 (d)) shows the Si-O-Al stretching [32]. The peaks at 668.55, 670.02, 617.89 and 660.10 cm^{-1}as shown in Fig. 4 (a), (b), (c) and (d) respectively, indicate the Si-O-Si bending. The Al-O stretching was also observed at 3649.11, 3628.10and 3635.03 cm^{-1} described in Fig. 4 (a), (b) and (d) respectively [33]. The systematic investigation about bonding information among different elements is shown in the Table 2.

So from the FTIR information it is clear that the oxidation is performed at the silicon surface, because the Si-O bonding information is present in the FTIR graph and Al-O bonding information is also present. So from SEM, EDX, OES and FTIR analysis it is clear that the oxidation is performed at the silicon surface in the argon oxygen plasma.

Table 2. FTIR spectroscopy analysis of the silicon Si (100).

	Si (100) samples which were exposed to argon plasma for			
	11 minutes	25 minutes	40 minutes	60 minutes
Si-O-Si bending	668.55 cm^{-1}	670.02 cm^{-1}	617.89 cm^{-1}	660.10 cm^{-1}
Si-O bending	809.60 cm^{-1}		818.51 cm^{-1}	805.29 cm^{-1}
Si-O-Al stretching	1091.76 cm^{-1}		1105.20 cm^{-1}	1112.35 cm^{-1}
Al-O stretching	3649.11 cm^{-1}	3628.10 cm^{-1}		3635.03 cm^{-1}

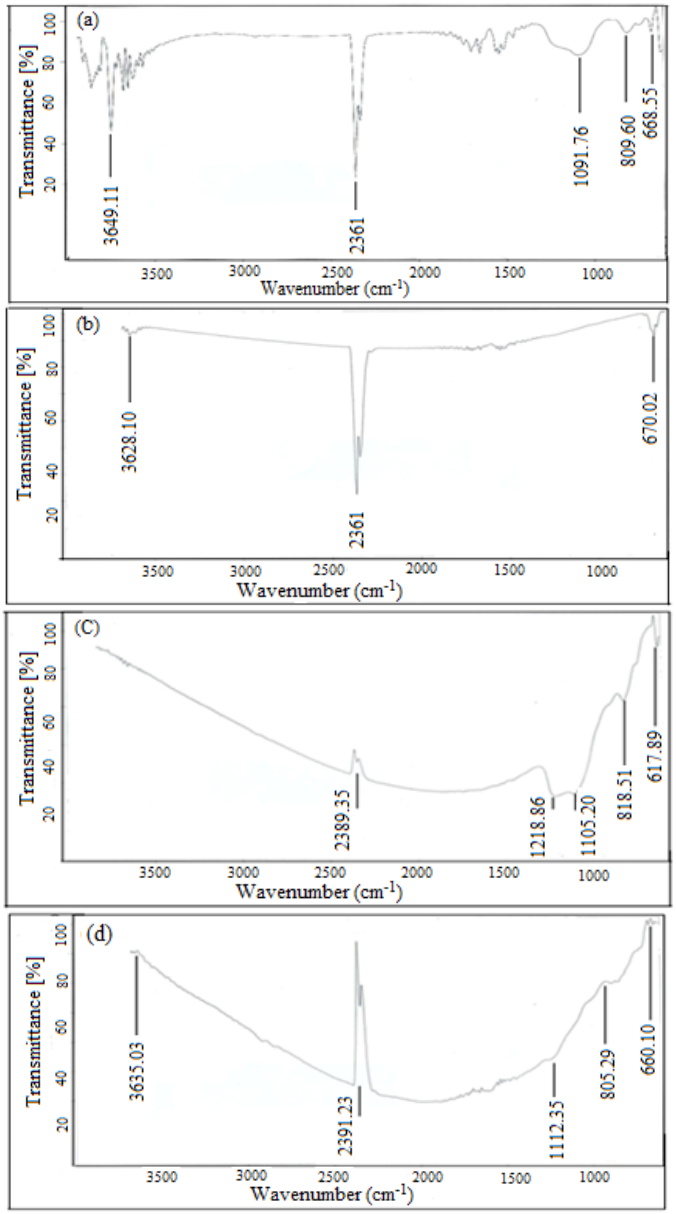

Fig. 4 FTIR graphs of the Si(100) after exposure to argon plasma for (a) 11 and (b) 25 min. (c) 40 and (d) 60 min.

3.3 Surface Modification of Aluminum (Al)

Now in this section the surface modification of aluminum Al is discussed. SEM micrograph and EDX graph of the Aluminum (Al) were taken before exposed to plasma not shown here. It is clear from the SEM micrograph the surface was plane and EDX graph conforms that only aluminum was present. For the investigation of the Al surface after exposed to plasma same techniques were used, which were used for silicon surface. The SEM microscopy of the Al samples which were prepared for 11, 25, 30 and 60 min by argon plasma as shown in the Fig. 5 (a), (b), (c) and (d) respectively. In these figures it can be clearly seen that the surface of aluminum has been modified. It is clearly obvious from SEM micrographs that when the plasma exposure time increases the layer of oxygen on the Al surface will increase. The SEM images were taken at the magnification of x 100 in Fig. 5 (a) and (b), but the SEM images were taken at the magnification x 300 for Fig. 5 (c) and x 600 for Fig. 5 (d). In the Fig. 5 (d) a small portion of the sample was focused, it is clearly obvious that the whole Al surface will be covered by the layer of the new material in that portion. So from the above discussion it is clear that a new material

(oxygen) layer has been formed on Al surface, for the conformation of the material the EDX analysis was also perform. EDX graphs of the same samples, which were prepared for 11, 25, 40 and 60 min were taken but only representative graph is shown in Fig. 6. In the graph there are two elements (Al and O) are present the substrate was of Al its means that layer of the new material is of oxygen.

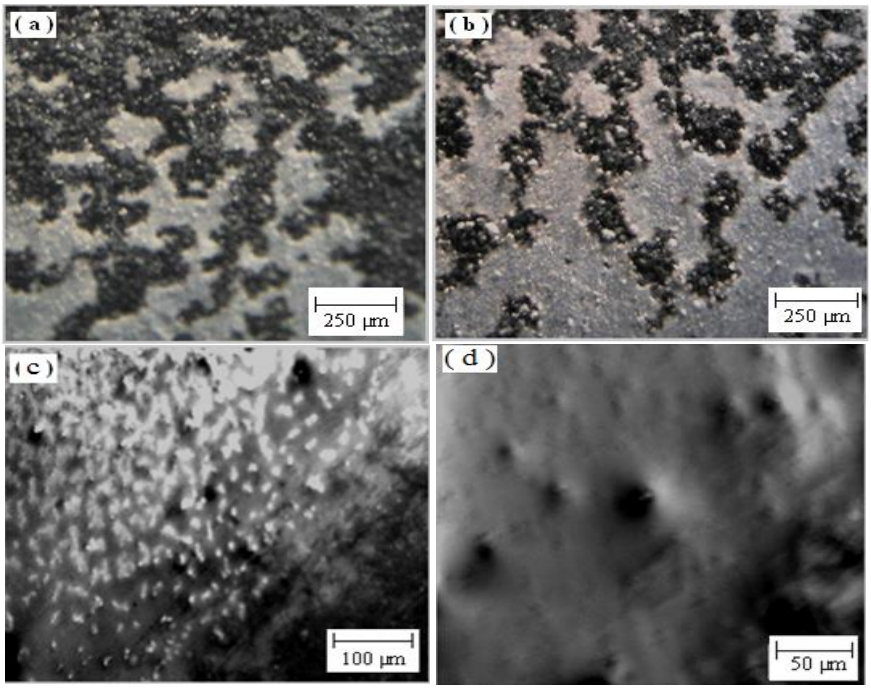

Fig. 5 SEM micrographs of Aluminum (Al) after exposure to argon plasma for (a) 11 (b) 25 (c) 40and (d) 60 min.

EDX also gives a semi quantitative analysis of the materials in terms of weight %. In the quantitative analysis the weight % of oxygen increases i.e 13.38% (for sample which was exposed by plasma for 11 min) to 17.35% (for sample which was exposed by plasma for 60 min). The quantitative analysis shows that the oxidation of Al increases when exposure time of plasma increases.

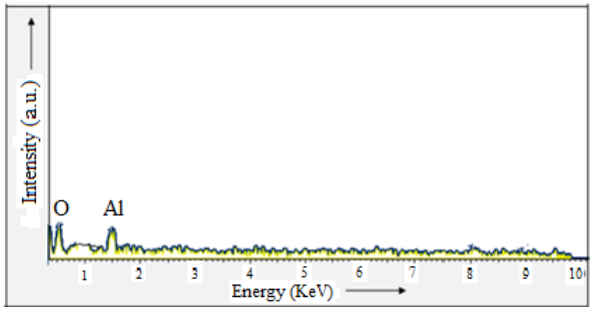

Fig. 6 EDX graph of aluminum (Al) after exposure to argon plasma for 40 min.

FTIR spectroscopy analysis was not performed for Al, because Al is not transparent for IR region. The cross sectional view of Si (100) and Al were also taken after exposed to plasma by SEM microscopy as shown in the Fig. 7 (a) and (b) respectively. The SEM micrographs show that the layer of oxygen has been formed on silicon (Si) and aluminum (Al) surfaces to form SiO_2 and Al_2O_3.

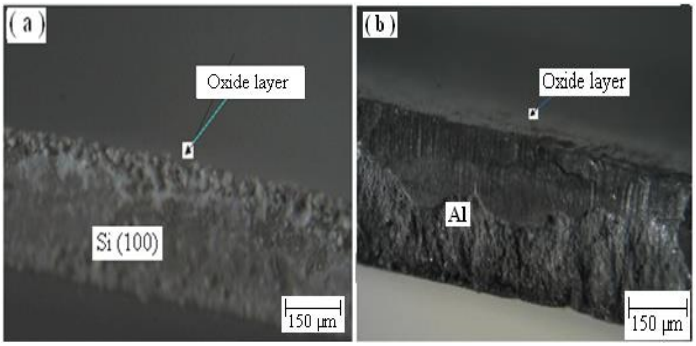

Fig. 7 Cross sectional view of SEM micrographs after exposure to argon plasma of (a) Si (100) and (b) Al.

4. Conclusions

APGD plasma was successfully generated by AC 50 Hz excitation of argon (Ar) inside a stainless steel chamber. Among all the atmospheric species, only oxygen was found to be activated. So plasma assisted oxidation of Si and Al surfaces was obtained selectively. Nature of gas, gas flow rate, electrode geometry, electrode spacing and applied electric field were found to be the key factor in sustaining a stable Ar/air discharge. Plasma electron temperature and electron number density, were calculated 0.56 eV (6500 k) and 1.7 x 10^{16} electrons par cm^3 respectively.

After successfully sustaining stable APGD plasma by 50 Hz AC power source, it was exposed to Si and Al surfaces for surface modification of the materials. It was found consistent with the observations that only oxidation of Si and Al was obtained. No nitriding or oxinitriding of the above said semiconducting and conducting materials were noted. Thus selective oxidation of Si and Al namely formation of SiO_2 and Al_2O_3 was successfully achieved. These materials can be used for biomedical applications.

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