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ORIGINAL ARTICLE

Influence of pre-treatment with non-thermal atmospheric pressure plasma on bond strength of TP340 titanium-PEEK direct bonding

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Abstract

Direct bonding of a TP340 titanium to PEEK by hot pressing via pre-treatment of non-thermal atmospheric pressure plasma jet has been demonstrated. The plasma irradiation effect on the bonding surface on the bond strength after hot pressing was investigated. The tensile shear strength of TP340-PEEK joined by hot pressing after plasma pre-treatment was measured by comparing specimens bonded using conventional hot pressing and those bonded using adhesives. The plasma treatment to the TP340 side resulted in the formation of TiO_2, which is chemically fed to oxide formation due to the irradiation of oxygen radicals generated by the plasma, resulting in a bond strength of less than 1 MPa, similar to the bond strength of the untreated specimens. The plasma irradiation effect on the PEEK side on the bond strength of TP340-PEEK bonded samples was also investigated. The bonding strength was increased by plasma irradiation to PEEK. As the plasma irradiation time was increased, the bonding strength gradually increased to 9.2 MPa, which is about 19 times higher than the bonding strength without plasma irradiation. These results suggest that oxygen radicals in the atmospheric pressure RF plasma jet produced oxygen-containing surface functional groups on the PEEK surface, which increased the strength of the TP340-PEEK direct joining.

Keywords Dissimilar material joining · Atmospheric pressure plasma · Direct joint

1 Introduction

Toward reducing global energy consumption, lightweight materials are being applied to reduce the weight of transportation equipment such as aerospace and automobiles [1]. One approach to weight reduction is the replacement of component materials with lighter aluminum, magnesium, and titanium, rather than steel, which has been the primary material used. The aerospace transportation equipment industry such as aerospace and automobiles has begun polymer materials using including carbon fiber-reinforced plastics, which have lightweight and high-strength properties to further reduce weight [2–5]. As mentioned above, light metals and metal-polymer hybrids are key materials owing to the requirement for lightweight design and integration of functions. Focusing on CFRP, the use of thermoplastic CFRP with high processability is expected to replace conventional thermosetting CFRP [6–9].

The advantage of using thermoplastic materials [10, 11] is that the materials can be joined directly by hot pressing using ultrasonic [12–15], induction [16], laser [17–22], or solid friction [23, 24] heat sources, eliminating the need for currently used bonding elements such as adhesives [25, 26], screws, or rivets [27, 28].

In general, it is known that the direct bonding mechanism between metal and polymeric materials is mainly hydrogen bonding between oxides on the metal surface and polar functional groups on the polymeric material surface [29–34]. Therefore, the addition of polar functional groups (carboxyl groups, amino groups, hydroxyl groups, etc.) on the surface of organic materials, along with the formation of a stable oxide layer on the metal surface, is very important for direct bonding. Therefore, in order to achieve reliable and high-strength direct metal–organic bonding, processes are needed to form oxide films on metal surfaces and to impart polar

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functional groups. Currently, for surface modification as pre-treatment for direct bonding by hot pressing, chemical etching using acids and alkalis [35], UV irradiation [36], corona treatment [37], and plasma treatment [38] are used. Plasma treatment is particularly promising in that it can efficiently modify only the surface of polymeric materials [38].

As one of the pre-treatment methods before hot pressing, an atmospheric pressure radio frequency (RF) plasma jet is proposed. The atmospheric pressure RF plasma jets allow the production of high-density oxygen radicals and the supplement of heat flux provided by the plasma (gas temperature around 150 °C) [39, 40]. Thus, the use of atmospheric pressure RF plasma jets has the potential to achieve efficient functional group impregnation because the radicals irradiated from the plasma and the surface heating from the incident heat flux from the plasma promote scientific reactions on metal and plastic surfaces.

Direct joining of SUS304 stainless steel and polycarbonate (PC), an engineering plastic with a low glass transition temperature, by combining surface treatment and heating with an atmospheric pressure RF plasma jet, without any heating source other than plasma has been demonstrated [41]. In addition, aluminum alloys A1050 and A5052 and the engineering plastic PEEK were directly joined by surface pre-treatment with atmospheric pressure RF plasma jets and thermocompression bonding to confirm the effect of plasma treatment on joint strength [42, 43].

In this paper, direct joining of pure titanium TP340 to polyetheretherketone (PEEK) with hot-pressing process using irradiation of atmospheric pressure plasma jet as a pre-treatment of bonding surfaces has been performed. The influence of plasma irradiation to TP340 and PEEK surfaces on the strength of TP340-PEEK bonding was investigated via observation of the chemical and physical state of the surfaces.

2 Experimental procedures

The sheets of pure titanium TP340 1.5 mm thick and PEEK (Mitsubishi Chemical Advanced Materials, Ketron 1000, melting temperature 340 °C) 5 mm thick, machined to a size of 50 × 15, were used as test pieces. Polyetheretherketone (PEEK), a semi-crystalline thermoplastic, exhibits excellent mechanical and chemical properties and high thermal stability, making PEEK widely used in various fields such as aerospace, automotive, and chemical process industries. PEEK has recently been studied and used as an alternative to metal implant materials due to its suitable biocompatibility and very low modulus (3–4 GPa) [44–48], reducing the degree of stress shielding often observed with titanium-based metal implants [49, 50].

As a plasma source for pre-treatment of direct bonding, an RF exited atmospheric Ar plasma jets as shown in Fig. 1a was used. The RF plasma jet consists of metal strips of lengths 15 mm and 5 mm, which were wrapped around a quartz tube to serve as the power and ground electrodes, respectively. The metal strip of 5 mm lengths as ground electrode was positioned at 2 mm from the top of the quartz tube, and the metal strip of 15 mm lengths as powered electrode was set 5 mm away from the under edge of the ground electrode. The dimensions of the quartz tube used for the plasma source are 6 mm outer diameter and 4 mm inner diameter. High-frequency (radio frequency: RF) power of frequencies 60 MHz was applied to the power electrode per matching network. The RF power of 78 W (V_{pp} = 1.6 kV) was applied to the powered electrode and Ar gas was supplied as the discharge gas at a gas flow rate of 3 slm [23, 24]. The ambient temperature and humidity, which are important parameters

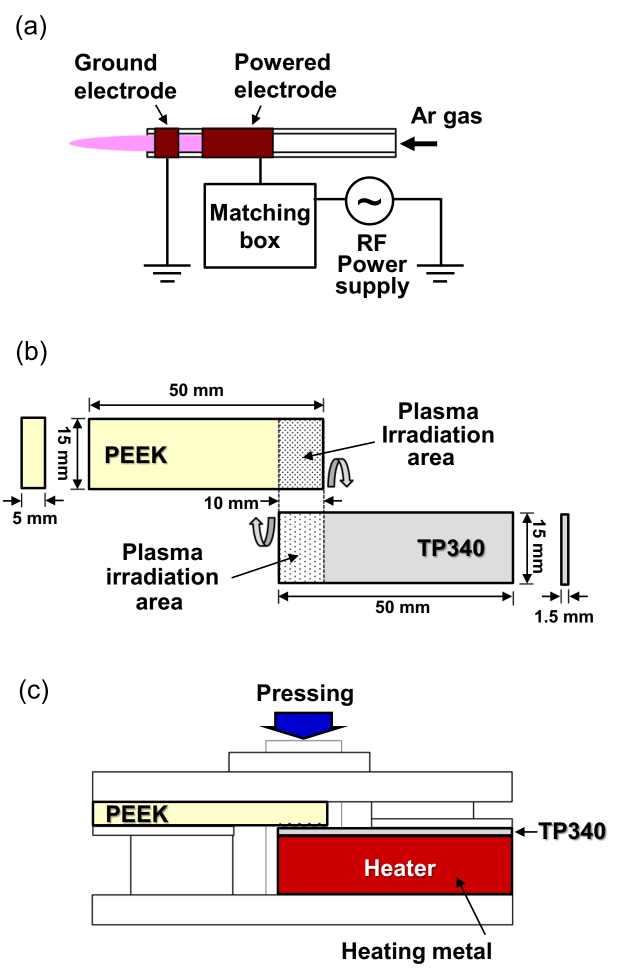

Fig. 1 Schematic illustrations of **a** apparatus used to generate an atmospheric pressure RF plasma jet, **b** dimensions of specimen, and **c** tensile shear configuration for TP340 and PEEK direct joining with hot-pressing process via pre-plasma treatment

during surface irradiation treatment with plasma [51], were approximately 20 °C and 40–60%, respectively.

Figure 1b, c shows the dimensions of the specimen and tensile shear configuration for direct joining of TP340 to PEEK using pre-treated samples by an atmospheric pressure RF plasma jet. As in the joining configuration shown in Fig. 1c, the TP340 and PEEK were joined with a 10 mm overlap. Figure 2 shows the procedure for TP340-PEEK direct joining based on atmospheric pressure RF plasma jet irradiation. The joining surface of TP340 and PEEK is subjected to surface treatment by plasma irradiation and then hot pressing as the direct bonding process was used.

Tensile shear strength of the TP340/PEEK direct joints were measured using the tensile shear tests (Autograph AGS-X: Shimadzu Corporation). The TP340 and PEEK sides direct joint specimens were clamped parallel to the axis of tension so that shear forces act on the bonded interface, and the maximum load at failure of the joint was measured at a crosshead speed of 1.66×10^{-3} mm/s.

The TP340 surface was analyzed using a scanning electron microscope (SEM; Hitachi SU-70) and its accompanying energy-dispersive X-ray spectrometer (EDX, Oxford Instruments INCA PentaFETx3). The surface morphologies of the TP340 were measured by atomic force spectroscopy (AFM) (KEYENCE VN-8000). Surface roughness in this study was estimated from an area of 10×10 μm in size, acquired by AFM.

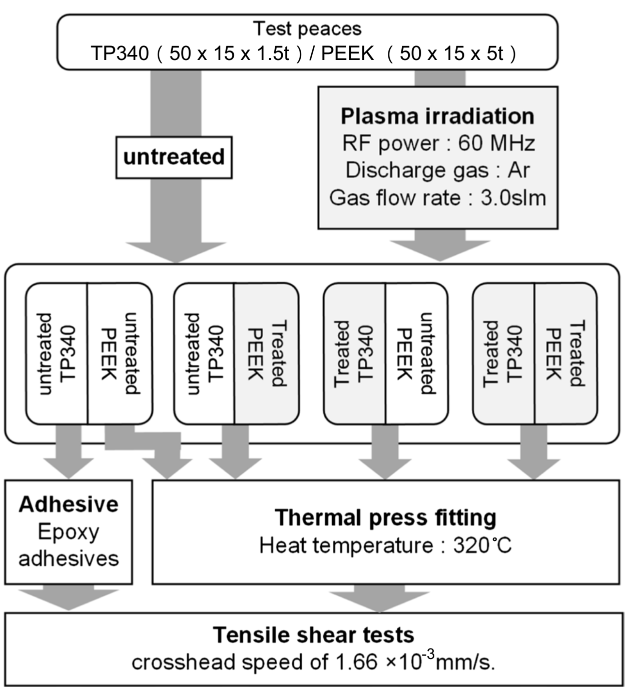

Fig. 2 Procedure for TP340-PEEK direct joining based on atmospheric pressure RF plasma jet irradiation

3 Results and discussion

An atmospheric pressure RF plasma jet, which shows the features such as the generation of a long plasma jet, 40 mm in length, is successfully and the operation in a non-thermal-equilibrium state at a gas temperature of around 300 °C [39], is used for the joints of metal and dissimilar materials in this study.

The plasma irradiation effects on the bonding strength of TP340-PEEK direct bonding have been investigated. TP340 and PEEK with and without plasma irradiation treatment were directly bonded by hot pressing. Figure 3 shows the tensile shear strength of specimen bonded by the hot pressing after the plasma treatment together with that of the hot pressing and adhesive joining using untreated TP340 and PEEK. The specimen bonded by hot pressing using untreated TP340 and PEEK have bond strength of 0.5 MPa. The plasma-irradiated Ti sample showed no increase in bond strength regardless of plasma irradiation of PEEK. In contrast, high-strength bonds of 9.2 MPa for samples which only PEEK was irradiated were obtained. This result suggests that atmospheric pressure RF plasma jet can be applied to the TP340-PEEK direct bonding. Figure 4 shows a photograph of the fractured surface after tensile testing of a specimen bonded with and without plasma irradiation. In the specimens irradiated with plasma on TP340, cohesive failure due to interfacial delamination and peeling of the weak layer on the surface of TP340 was observed regardless of whether the specimens were irradiated with or without plasma on

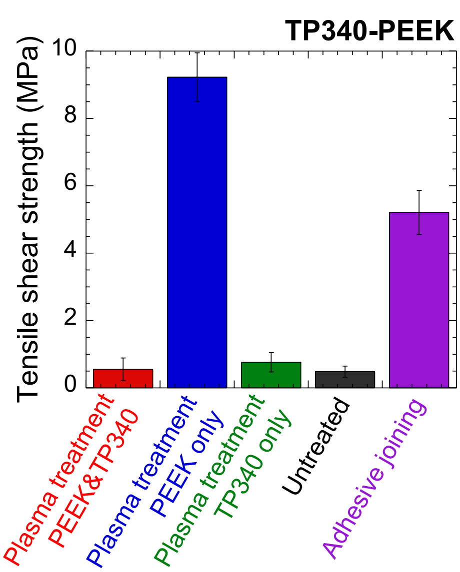

Fig. 3 Tensile shear strength of specimens bonded by hot press method using TP340 and PEEK pretreated with an atmospheric pressure RF plasma jet and untreated TP340 and PEEK together with that of samples bonded with adhesive using untreated TP340 and PEEK

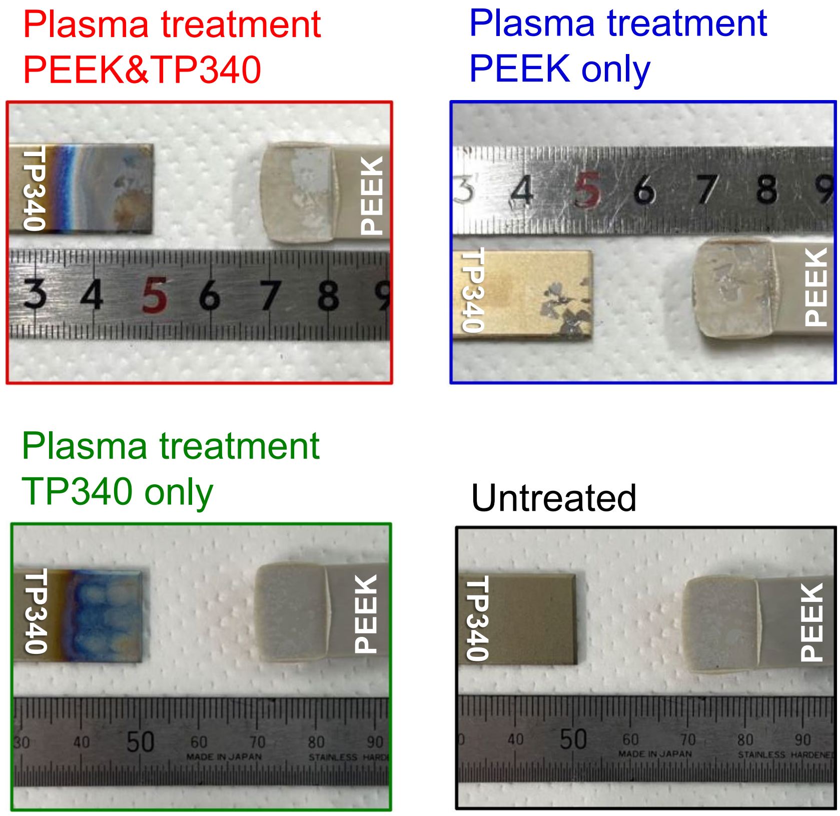

Fig. 4 Photographic images of fracture surfaces after tensile testing of specimens bonded with and without plasma irradiation

PEEK. On the other hand, the specimens bonded by plasma irradiation to PEEK showed interfacial delamination and cohesive failure of PEEK due to strong bonding between TP340 and PEEK. Some complete interfacial delamination was also observed in the specimens bonded with untreated TP340/PEEK.

In direct bonding of polymers and metallic dissimilar material, the physical and chemical condition of surfaces is known to affect the bonding strength. The effect of plasma irradiation on the physical and chemical surface conditions was investigated, focusing on TP340, which affects the bonding strength due to plasma irradiation. For investigating the physical effects of plasma irradiation on the TP340 surface, the surface morphology of the TP340 surface after plasma irradiation was measured by using AFM. Figure 5 shows the surface roughness estimated by the AFM observation of TP340 surface treated at different plasma irradiation time. When the plasma irradiation time was increased, the surface roughness R_a of the TP340 surface increased slightly and then was almost constant at 400 ~ 450 nm as shown in Fig. 5. It is generally known that surface roughness affects joint strength due to the anchoring effect. Therefore, this result suggests a physical influence on bonding strength due to the relatively large surface roughness over 400 nm.

Change in the chemical composition on TP340 surface due to plasma irradiation was measured by SEM–EDX. Figure 6 shows top surface SEM images and SEM–EDX elemental maps of Ti (yellow), O (red), and Fe (green) obtained from the TP340 surface with and without plasma exposure. The SEM image after the plasma irradiation shows that there is no significant change in the surface roughness, but the formation of grain structure seen during general high-temperature oxidation [52] is observed. Therefore, these SEM images indicate that minimal physical change was induced by irradiation. On the other hand, the SEM–EDX elemental map of O shows drastic

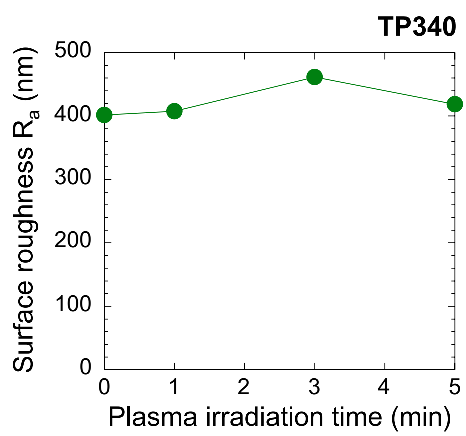

Fig. 5 Variation of surface roughness R_a of TP340 on plasma irradiation time

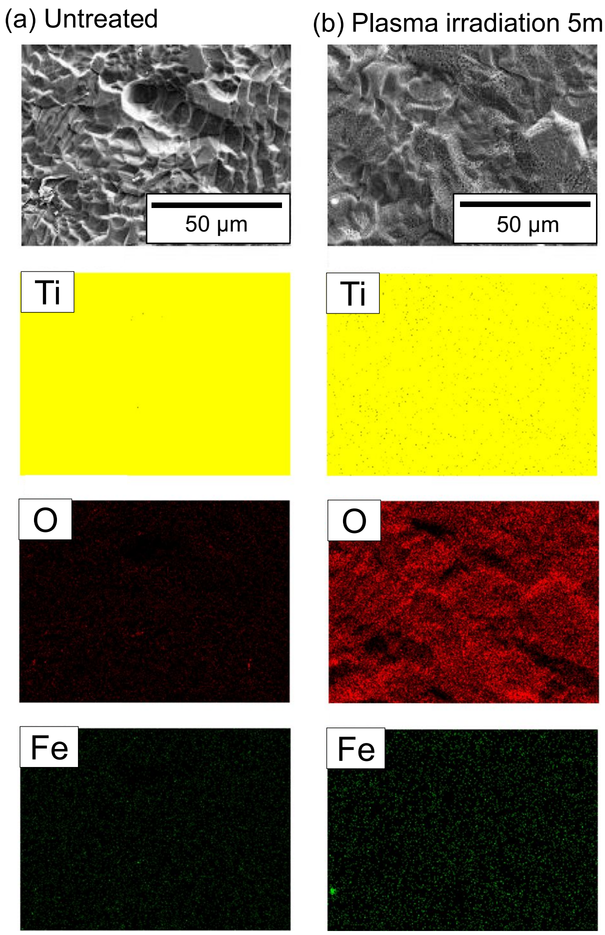

Fig. 6 SEM images and SEM–EDX elemental maps of Ti (yellow), O (red), and Fe (green) on surface of **a** untreated TP340 and **b** TP340 exposed to plasma for 5 min

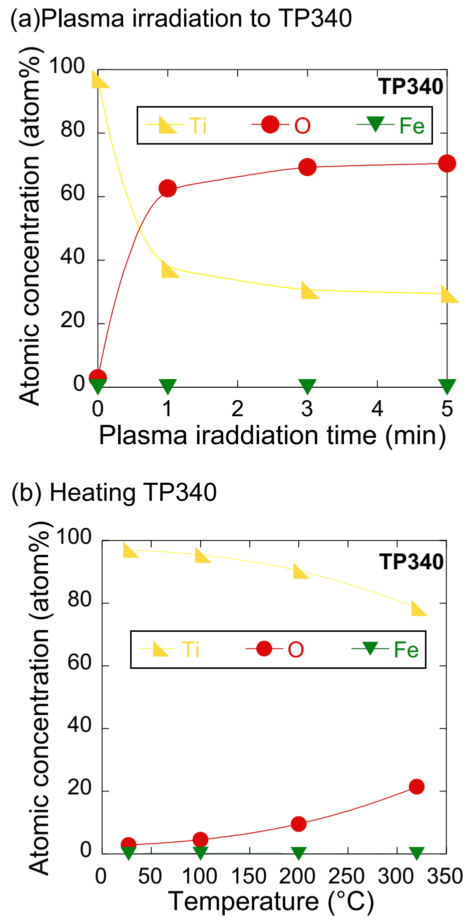

Fig. 7 Atomic concentration of TP340 surface **a** as a function of plasma irradiation time and **b** as a function of surface temperature heated by thermal heating only

oxidation after plasma treatment. Figure 7 summarizes the atomic concentration of Ti, O, and Fe estimated from SEM–EDX elemental maps when the plasma irradiation time was varied (Fig. 7a), compared with oxidation behavior due to thermal heating only (Fig. 7b). With increasing plasma irradiation time, the atomic concentration of O on the TP340 surface considerably increases about 60 at% for plasma irradiation time of 1 min and then slightly increases to 66 at% for 5 min. On the other hand, in the case of thermal oxidation in air, the oxygen concentration only increased to about 21 at% even when the Ti specimen was heated to 320 °C. This result shows that oxidation by plasma irradiation is sufficiently advanced compared to oxidation by heating during direct bonding. The increase in the relative concentration of oxygen is considered to be caused by the enhancing oxidation on TP340 surface at low temperature compared to oxidation by heat treatment due to the synergistic effect of the surface heating to about 300 °C by the heat flux from an atmospheric pressure RF plasma jet and irradiation of oxidation species including O and OH radicals generated by an atmospheric pressure RF plasma jet. Therefore, a strong oxide film is formed on the Ti surface to inhibit oxidation into the Ti interior, and little solid dissolution of oxygen into the Ti interior can be expected to proceed, thus preserving the raw material state in the bulk [53]. Generally, TiO_2 films formed during oxidation by heat treatment in an atmospheric atmosphere have a multilayer structure with cracks, micro-voids, and partial delamination, which is caused by compressive stresses in the TiO_2 film due to the volume expansion caused by oxidation. The volume ratio of Ti to TiO_2 (Pilling-Bedworth ratio) is 1.8, which is an index of stress generated by oxidation [54]. In this study, the SEM–EDX results show that the surface of the Ti specimen immediately after plasma irradiation is sufficiently oxidized to be close to TiO_2 in stoichiometric composition, but no peeling off of the TiO_2 surface was observed immediately after plasma irradiation. When this specimen was bonded to PEEK after plasma irradiation treatment by hot pressing and then subjected to a tensile test, the

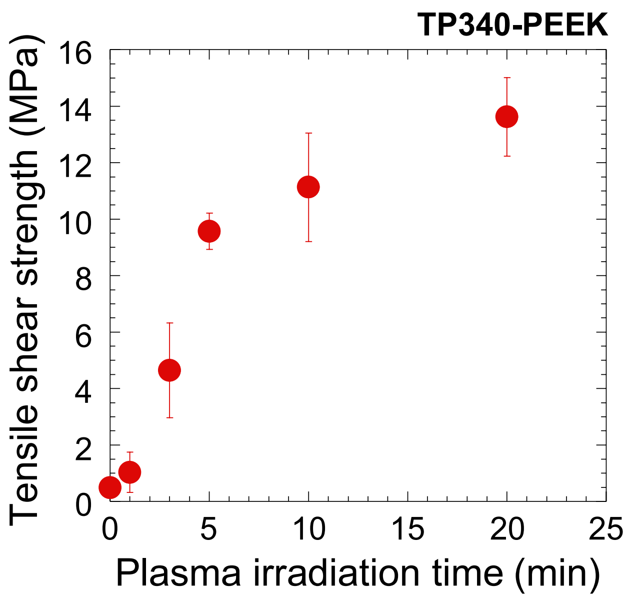

Fig. 8 Variation of tensile shear strength of TP340-PEEK bonded samples following plasma treatment only of PEEK side with plasma irradiation time

film peeled off from the Ti specimen side and remained on the bonding surface on the PEEK side. These results indicate that TP340 can be sufficiently oxidized at low temperatures by reactive oxygen species generated by the plasma jet.

Figure 8 shows the change of tensile shear strength of TP340-PEEK direct bonded samples with no treatment applied to the TP340 when the plasma irradiation time to PEEK was changed. With increasing plasma irradiation time, the bond strength increased significantly from 0.48 MPa at 0 min to 9.57 MPa at 5 min, and then slowly increased to 13.6 MPa at 20 min. The effect of plasma irradiation on PEEK has already been investigated in previous studies [41]. The results of surface analysis by XPS confirmed that the O-C = O bonds were formed by plasma irradiation in addition to the C-O and C = O bonds that were originally present in PEEK. The amount of these groups attributed to oxidation on the PEEK surface is found to increase with plasma irradiation time. In general, O = C-O groups formed on polymers are known to increase the bond strength between metal and polymer following direct bonding [55–57]. Thus, these results suggest that oxidation of PEEK by oxygen radicals in an atmospheric pressure RF plasma jet produced oxygen-containing surface functional groups that increased bond strength. Thus, these results suggest that the increase in oxygen-containing surface functional groups due to oxidation of PEEK by radicals in atmospheric pressure RF plasma jets increases bond strength of TP340-PEEK direct joining.

4 Conclusions

Direct bonding of a TP340 to PEEK by hot pressing via pre-treatment of non-thermal atmospheric pressure plasma jet has been demonstrated. The plasma irradiation effect on the bonding surface on the bond strength after hot pressing was investigated. The tensile shear strength of TP340-PEEK joined by hot pressing after plasma pre-treatment was measured by comparing specimens bonded using conventional hot pressing and those bonded using adhesives. The tensile shear stress of the direct bonded sample by plasma irradiation to PEEK only was 9.2 MPa, 184% higher than 0.5 MPa strength of the unirradiated sample. On the other hand, plasma treatment to the TP340 side resulted in a decrease in bond strength as a result of TiO_2 formation, which is easily chemically fed to form oxides by irradiation of oxygen radicals generated from plasma. This reduction in bond strength is considered to be due to the fact that TiO_2 films formed by oxidation by plasma treatment tend to delaminate at the Ti/TiO_2 interface because the volume expansion caused by rapid oxidation generates compressive stress in the TiO_2 film.

The plasma irradiation effect on the PEEK side on the bond strength of TP340-PEEK bonded samples was also investigated. As the plasma irradiation time increased, the bond strength increased significantly from 0.48 MPa for 0 min to 9.57 MPa for 5 min, then slowly increased to 13.6 MPa for 20 min, a 283% increase in bond strength over untreated. These results suggest that oxygen radicals in the atmospheric pressure RF plasma jet produced oxygen-containing surface functional groups on the PEEK surface, which increased the strength of the TP340-PEEK direct joining.

Author contribution Kosuke Takenaka contributed to all parts of this work: conceptualization, investigation, and writing the manuscript. Soutaro Nakamoto, Ryosuke Koyari, Akiya Jinda, and Susumu Toko contributed to collecting and analyzing the data of surface measurement and the joining experiments. Giichiro Uchida contributed to assistance of conceptualization, analyzing the data of investigation. Yuichi Setsuhara contributed to conceptualization, supervision, and project administration. All authors read and approved the final manuscript.

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Declarations

Ethics approval Not applicable.

Consent to participate Not applicable.

Consent for publication The authors give the publisher the consent to publish the work.

Competing interests The authors declare no competing interests.

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