The relation between residual stress, interfacial structure and the joint property in the SiO$_2$f/SiO$_2$-Nb joints

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In order to achieve a high-quality joint between SiO$_2$f/SiO$_2$ and metals, it is necessary to address the poor wettability of SiO$_2$f/SiO$_2$ and the high residual stress in SiO$_2$f/SiO$_2$-Nb joint. Here, we simultaneously realize good wettability and low residual stress in SiO$_2$f/SiO$_2$-Nb joint by combined method of HF etching treatment and Finite Element Analysis (FEA). After etching treatment, the wettability of E-SiO$_2$f/SiO$_2$ was improved, and the residual stress in the joint was decreased. In order to better control the quality of joints, efforts were made to understand the relationship between surface structure of E-SiO$_2$f/SiO$_2$ and residual stress in joint using FEA. Based on the direction of FEA results, a relationship between residual stress, surface structure and joint property in the brazed joints were investigated by experiments. As well the FEA and the brazing test results both realized the high-quality joint of E-SiO$_2$f/SiO$_2$-Nb and the shear strength of the joint reached 61.9 MPa.

SiO$_2$/SiO$_2$, one of the most significant functional and structural quartz fiber reinforced silica ceramic matrix composites, has been attracted great attention in aerospace industry applications, due to its high thermal shock resistance, excellent ablation resistance as well as low thermal conductivity$^{1-6}$. However, like most advanced ceramic matrix composites, it is difficult for SiO$_2$/SiO$_2$ to fabricate large-sized or complex-shaped components owing to its intrinsic brittleness and low fracture strain$^{7-12}$. Therefore, it is indispensable to develop a reliable technique to joint SiO$_2$/SiO$_2$ with metals to extend the application of the composite.

Due to the intrinsic brittleness of SiO$_2$/SiO$_2$, usually, fusion welding is not applied because of the possibility of brittle fracture forming during cooling$^{13}$. Alternatively, although adhesive bonding can be performed to realize SiO$_2$/SiO$_2$-metal joint, the strength of the joint reduce during long service. Consequently, vacuum brazing is the primary method to joining SiO$_2$/SiO$_2$ to metals because of its cost-effectiveness and high-quality process$^{14-22}$. However, two crucial challenges: the poor wettability of SiO$_2$/SiO$_2$ and the high residual stress in the joint exist in brazing SiO$_2$/SiO$_2$ to metal, which seriously impairs the strength of the joints. In view of that, some researchers demonstrated that the wettability of SiO$_2$/SiO$_2$ with active brazing alloy can be successfully improved by coated nickel$^{23}$, CaCO$_3$$^{24}$, or few-layer graphene$^{25}$. Although the wettability of SiO$_2$/SiO$_2$ with active brazing alloy is improved, a high-quality joint is difficult to obtain because of the negligence to the high residual stress.

So far, by micro-machine process on the surface of ceramic or composite, forming a 3D composite-metal gradient transition zone in their joints has been developed to reduce the residual stress of the brazed joints$^{26-28}$. Zhang et al.$^{26}$ demonstrated that fabricating microscale periodic surface pattern on Al$_2$O$_3$ ceramic surface was a promising method to form a 3D transition region and reduce the residual stress of ceramic-metal joints. Wang et al.$^{27}$ reported that drilling holes on the surface of C/C composite can reduce the residual stress because of constructing a 3D C/C-metal gradient transition zone in joint. Shen et al.$^{28}$ demonstrated that drilling blind holes on C/C composite surface by means of laser can result in significantly strengthening and toughening the joint due to creating a 3D transition region between the C/C and braze. To some extent, micro-machining on its surface can lead to damaging the whole structure of SiO$_2$/SiO$_2$ due to its intrinsic brittleness and braided structure. Undoubtedly, it is urgently to provide an effective method to both improving the wettability with active brazing alloy and forming a 3D gradient transition zone at SiO$_2$/SiO$_2$ side without impairing the overall structure.
Our latest work showed that a 3D-pinning structure was beneficial to the joint strength. However, the structure introduced the residual stress among the braided quartz fibers, which complicated the distribution of the residual stress in the joint. Furthermore, the complex structure of the 3D SiO$_2$/SiO$_2$-metal gradient transition zone had a decisive influence on the mechanical property of joint. Thus, the relationship between the residual stress, the surface structure and joint property in the brazed joints need be explored in depth and in detail.

In this paper, the etching treatment with HF acid solution was designed to regulate the surface structure of SiO$_2$/SiO$_2$. Finite element analysis (FEA) models were developed to study the relationship between the residual stress and the surface structure in the brazed joints, which can guide the subsequent experiment and provide the theoretical basis. Furthermore, the relationship between the residual stress, the surface structure and the joint property in the brazed joints was discussed in detail.

**Experimental procedures**

**Materials.** The 3D four-directional braided SiO$_2$/SiO$_2$ and commercially available Nb were used as the parent materials. The dimension of SiO$_2$/SiO$_2$ brazing specimen was 5 mm $\times$ 5 mm $\times$ 3 mm. Nb was cut into 10 mm $\times$ 10 mm $\times$ 3 mm slices for the microstructure observation and 10 mm $\times$ 15 mm $\times$ 3 mm for shear tests, respectively. The active brazing alloy foil Ag-21Cu-4.5Ti (wt.%) with a thickness of 200 $\mu$m was used to braze SiO$_2$/SiO$_2$ and Nb. The bonding surfaces of samples were ground up to 400 by SiC sandpaper. All materials were ultrasonically cleaned in acetone for 15 min.

The etching treatment on the surface of SiO$_2$/SiO$_2$ was performed with 20 wt.% HF acid solution. The schematic of the etching process is shown in Fig. 1. Firstly, the HF acid solution was directly placed on the surface of SiO$_2$/SiO$_2$ for a few seconds. Then, the surface was washed with deionized water carefully. Finally, SiO$_2$/SiO$_2$ with etching treatment (E-SiO$_2$/SiO$_2$) was obtained. In addition, by controlling etching process, the surface structure dimension can be tunable.

**Wetting and brazing processes.** Wetting experiments were performed using the sessile drop technique in which the alloys are placed on the substrate and the system is heated to 840 °C. For comparison, during the sessile drop experiments, AgCuTi brazing alloy foils were placed on SiO$_2$/SiO$_2$ with different infiltration depth, respectively. And the brazing experiments were performed with AgCuTi active brazing alloy foils between the parent materials. The structure of the assembly was SiO$_2$/SiO$_2$/AgCuTi/Nb, and the assemblies were held by graphite jigs. In order to keep the specimens in close contact, a load of 0.01 MPa was applied. The assemblies were heated to 840°C with a rate of 10 °C min$^{-1}$ in a vacuum furnace, isothermally held for 10 min, and then cooled down to room temperature at a rate of 5 °C min$^{-1}$.

The drop images, which were produced by an optical system coupled with a zoom (magnification 30×), were recorded by a video camera connected to a computer, permitting automatic image analysis. This device enables the contact angles of the drop were measured with an accuracy of ±2°. The interfacial microstructures of the joints were analyzed by a scanning electron microscopy (SEM) fitted with an energy dispersive spectroscopy (EDS). To identify the phases formed in the reaction layers adjacent to SiO$_2$/SiO$_2$ and fracture, a JDX-3530M X-ray diffraction (XRD) was used. To evaluate the mechanical properties of the joints, shear tests were carried out using an Instron-1186 universal testing machine at room temperature. The average stress strength was identified by five shear specimens brazed under the same condition.

**FEA calculations.** The FEA method was employed to investigate the distribution of the residual stress along the SiO$_2$/SiO$_2$-Nb brazed joint in our research, because the method was proved to be a useful tool for predicting residual stress in the joint$^{26,31}$. Thus, in this paper, the distribution of the residual stress, which yielded in the
joint during cooling due to the mismatch of Coefficient Thermal Expansion (CTE), was simulated by FEA with Marc-2013.

Results and Discussion

Microstructure of the joint brazed with and without etching treatment. The interface analysis was first performed on the brazing joints before and after etching treatment, evidencing the establishment and the application of model for FEA. The typical microstructure of SiO$_2$/SiO$_2$-Nb joint is shown in Fig. 2a. It can be found cracks formed at SiO$_2$/SiO$_2$ side, which the typical interface structure of the joint was SiO$_2$/SiO$_2$/Ag$_{(s,s)}$/Cu$_{(s,s)}/$Cu$_2$Ti$_3$O$_7$/TiSi$_2$, based on our latest research. And the mismatch of CTE between SiO$_2$/SiO$_2$ (CTE$_{SiO2/SiO2}$ = $~2.0 \times 10^{-6}$/K) and Nb (CTE$_{Nb}$ = $~7 \times 10^{-6}$/K) or AgCuTi active brazing alloy (CTE$_{AgCuTi}$ = $~15.4 \times 10^{-6}$/K) is high, which results in forming cracks. Figure 2b shows the typical microstructure of E-SiO$_2$/SiO$_2$-Nb joint. Compared with SiO$_2$/SiO$_2$-Nb joint, the primary compositions of beam were nearly same. The brazing alloy infiltrated into E-SiO$_2$/SiO$_2$ and formed a “3D-pinning structure”, which contributed to form a good CTE gradient transition and to reduce its mismatch between different materials. Consequently, the joint exhibited sound bonding without any defect and crack.

In order to further investigate the interfacial microstructure of the E-SiO$_2$/SiO$_2$-Nb joint, the main elements distribution of the joint produced at 840 °C for 10 min are analyzed, as shown in Fig. 3. The Fig. 3a clearly presents that a sound joint has been obtained. Figure 3b–f shows the distribution of Ag, Cu, Ti, Si and Nb, respectively. It can be seen that the Si had a strong tendency to extremely react with Ti, as shown in Fig. 3d and e. In addition, notice that Ti-rich granular were formed adjacent to SiO$_2$/SiO$_2$/Ag$_{(s,s)}$/Cu$_{(s,s)}/$Cu$_2$Ti$_3$O$_7$ composite, revealed that Ti segregated in 3D SiO$_2$/SiO$_2$-metal gradient transition zone. Furthermore, the distribution of Ti in that zone was not even because the brazing alloy gradually infiltrated into E-SiO$_2$/SiO$_2$ and Ti reacted with the contacted quartz fibers. Thus, the remaining Ti became less and less as the infiltration depth increasing. Moreover, it was important to note that SiO$_2$/SiO$_2$ and Nb did not spread or dissolve during brazing, as shown in Fig. 3b and f, respectively. Therefore, based on the above results, the model for FEA can be developed as three parts: SiO$_2$/SiO$_2$ (or E-SiO$_2$/SiO$_2$), AgCuTi brazing alloy and Nb (see Fig. 4). It was worth noting that the special structure of the E-SiO$_2$/SiO$_2$ by the etching treatment needed a completely new design system (Details on the model were shown in supplementary material).

Estimation of residual stress in the brazed joint using FEA. Recently, many researches have focused on reducing the residual stress in the composite/ceramic and metal brazed joints. In fact, it is very difficult to measure the residual stress of the brazed joints directly through the experimental measurement. Thus, it is general to analyze the residual stress of the brazed joints from the joint fracture path. However, this analysis only horizontal contrast (trend), cannot be quantified (experimental value) contrast. In addition, when the fracture path exists in the same area, it is difficult to analyze the residual stress of composite/ceramic and metal brazed joints. In order to analyze the residual stress variation better, some researchers always investigated the distribution of residual stress by Finite Element Analysis (FEA). In our case, the fracture path is different before and after etching treatment. Especially, after etching treatment, fractures all exist close to the etching area, then it is not accurate to analyze the residual stress through the fracture path. Therefore, based on the typical experimental results, the values of residual stress of the samples with varied etching depth can be examined by FEA method.

Based on the analysis in 3.1 section, the FEA was applied to simulate the distribution of residual stress in the brazed joint, and the details on simulation process were shown in supplementary material. The etching depth was related to the dimension of “3D-pinning structure” which directly affected the residual stress in the joint. Therefore, it is important to investigate the relationship between the surface structure (that is the layer thickness of 3D-pinning structure) and the residual stress in a brazed joint, which can provide the theoretical basis for the following brazing experiments.

Figure 5 shows the distribution of equivalent von mises stress in 0μm@E-SiO$_2$/SiO$_2$-Nb (denoted as “Xμm@E-SiO$_2$/SiO$_2$-Nb for convenience, X represented the etching depth), 50μm@E-SiO$_2$/SiO$_2$-Nb, 75μm@E-SiO$_2$/SiO$_2$-Nb, 100μm@E-SiO$_2$/SiO$_2$-Nb, 125μm@E-SiO$_2$/SiO$_2$-Nb and 150μm@E-SiO$_2$/SiO$_2$-Nb joints.
joints brazed by AgCuTi at 840 °C for 10 min. It is obvious that the residual stress has been gradually changed with the etching depth increasing from 0 μm to 150 μm. As for 0 μm@E-SiO₂f/SiO₂-Nb joint (that is SiO₂f/SiO₂-Nb joint), the residual stress is constrained around the 0 μm@E-SiO₂f/SiO₂-AgCuTi interface, and the peak residual stress of 380 MPa (see Fig. 6) generates in the SiO₂f/SiO₂ side close to brazing alloy, and then gradually decreased along the vertical direction of 0 μm@E-SiO₂f/SiO₂-AgCuTi interface. After etching treatment, the higher residual stress in E-SiO₂f/SiO₂-Nb joints has transferred in the “3D-pinning structure”, which suggested that the structure played a key role in the distribution of the residual stress in the joints (see Fig. 5b–f). Moreover, it is worth noting that the maximum residual stress of E-SiO₂f/SiO₂-Nb joints has transferred on the braided quartz fibers in the “3D-pinning structure”, as shown in Fig. 5b–f. Furthermore, it clearly presents that the residual stress in the joints and on the braided quartz fibers were both reduced with the etching depth increasing. However, when the etching depth was too thick, the residual stress increased significantly, especially on the braided quartz fibers, as shown

**Figure 3.** Interfacial microstructure and elemental distribution of E-SiO₂f/SiO₂-Nb joint (a) BSE image of the joint and EDS maps of (b) Ag, (c) Cu, (d) Ti, (e) Si and (f) Nb.

**Figure 4.** Mesh of finite elements for (a) the brazed joint, (b) magnification of the E-SiO₂f/SiO₂, (c) magnification of AgCuTi brazing alloy.
From the above results, it can be inferred that the “3D-pinning structure” can effectively reduce the residual stress and change the distribution of residual stress in the joints.

As for the 0μm@E-SiO$_2$/SiO$_2$-Nb joints, the high CTE mismatch may cause the residual stress constraining around the SiO$_2$/AgCuTi interface. After etching treatment, a “3D-pinning structure” formed in the joint, which was contributed the brazing alloy infiltrating into the E-SiO$_2$/SiO$_2$ side, forming a 3D SiO$_2$/metal gradient transition zone. The zone was beneficial to reduce the residual stress induced by the high mismatch of the dissimilar substrates. However, the zone also made the distribution of the residual stress in “3D-pinning structure” complicated, as shown in Fig. 5. In particular, when the etching depth further increased, the residual stress rather than reduced. In order to analyses the reason, we explored the relationship between the residual stress of joint in different directions and surface structure of the E-SiO$_2$/SiO$_2$ by FEA. The schematic diagram of the profile of the model used in simulation procedure is shown in Fig. 7a. According to the structure of the E-SiO$_2$/SiO$_2$-Nb joints and our calculation results, it can be inferred that principal stress $\sigma_z$ changed with the etching depth increasing, but $\sigma_x$ and $\sigma_y$ did not or very little change. Another significant stress was shear stress $\tau_{xy}$, which latter, in combination with $\sigma_z$, can induce fracture of the quartz fibers. In addition, shear stress $\tau_{xz}$ and $\tau_{zy}$ changed very little with the etching depth increasing. So, the following analysis only focused on the largest principal stress $\sigma_z$ and shear stress $\tau_{xy}$ in E-SiO$_2$/SiO$_2$-Nb joints. Figure 7b and c show the maximal $\sigma_z$ and $\tau_{xy}$ in zone A of E-SiO$_2$/SiO$_2$-Nb joints, respectively. It can be observed that after etching treatment, $\sigma_z$ reduced markedly (from 0 to 100μm), but a little varied with the etching depth increasing to 150μm, as shown in Fig. 7b. In contrast, it is noteworthy that $\tau_{xy}$ significantly increased with the etching depth further increasing (from 100 to 150μm), as shown in Fig. 7c. Thus, the maximal resultant force in the zone descended first (from 0 to 100μm) and then ascended (from 100 to 150μm), as shown in Fig. 6. From the above results, it can be concluded that with the

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**Figure 5.** Distribution of equivalent von mises stress in (a) 0μm@E-SiO$_2$/SiO$_2$-Nb, (b) 50μm@E-SiO$_2$/SiO$_2$-Nb, (c) 75μm@E-SiO$_2$/SiO$_2$-Nb, (d) 100μm@E-SiO$_2$/SiO$_2$-Nb, (e) 125μm@E-SiO$_2$/SiO$_2$-Nb and (f) 150μm@E-SiO$_2$/SiO$_2$-Nb joint.

**Figure 6.** Comparison of equivalent von mises stress for different etching depth.

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etching depth between 0 to 100 μm, the residual stress of the joint can be reduced, because of the formed 3D SiO₂f/SiO₂-metal gradient transition zone, which was contributed to decrease the CTE mismatch. Nevertheless, with the etching depth further increasing (from 100 to 150 μm), the residual stress was no lower, but higher, due to the τₓᵧ which was introduced by the zone and posed serious problems for the joint. Therefore, it can be concluded that although the “3D-pinning structure” can reduce the residual stress, the dimension of the structure should be in proper domain.

Effect of surface structure on the wettability of SiO₂f/SiO₂ composite. It is well known that the wettability of SiO₂f/SiO₂ plays an important role in obtaining a high-quality brazing joint. Therefore, it is necessary to investigate the effect of etching treatment on the wettability of AgCuTi brazing alloy on SiO₂f/SiO₂ surface. Figure 8 shows the contact angles (CA) of AgCuTi brazing alloy on the surface of 0 μm@E-SiO₂f/SiO₂, 50 μm@E-SiO₂f/SiO₂, 75 μm@E-SiO₂f/SiO₂, 100 μm@E-SiO₂f/SiO₂, 125 μm@E-SiO₂f/SiO₂ and 150 μm@E-SiO₂f/SiO₂, respectively. It can be observed that the brazing alloy on SiO₂f/SiO₂ shows unsymmetrical round shape, with left side showing smaller contact angles compared to the right one. And there are two main reasons for it. Firstly, the SiO₂f/SiO₂ brazing specimen was obtained through cutting into slices and grounding the bonding surface. So, after that treatment, the bonding surface of SiO₂f/SiO₂ was not flat, which led to the surface of E-SiO₂f/SiO₂ uneven, even the slope. Secondly, the surface stability of liquid brazing alloy on E-SiO₂f/SiO₂ was affected in vacuum furnace by mechanical pump and molecular pump during vacuumizing. Therefore, the brazing alloy on the surface of E-SiO₂f/SiO₂ showed unsymmetrical round shape in Fig. 8. However, the left contact angles (CA) of AgCuTi brazing alloy on SiO₂f/SiO₂ decreased from 138° to 53° as shown in Fig. 8b. The CA decreased with the etching depth increasing, though the brazing alloy on SiO₂f/SiO₂ shows unsymmetrical round shape. Then, in our case, the right CA acted as the evaluation standard.

Figure 7. (a) Schematic diagram of model and coordinate half system adopted in calculation and residual stress distribution of SiO₂f/SiO₂ side closed to interface (b) σₓᵧ (c) τₓᵧ.
the infiltration depth was gradually increased with etching depth increasing, as shown in Fig. 9. After etching treatment, only the silica sol was consumed and numerous quartz fibers were left on the surface of SiO$_2$/SiO$_2$\textsuperscript{17}. Based on the above results, we believe that the poor wettability of SiO$_2$/SiO$_2$ can be owing to the silica sol, and the wettability between brazing alloy and quartz fibers was extremely well. It suggested that the etching treatment was an easy and effective way to improve the wettability of SiO$_2$/SiO$_2$. Thus, it can be inferred that the space of the consumed silica sol was able to be filled up with sufficient brazing alloy.

Generally, the wettability of the materials with the same surface state is almost identical under the same condition. The surface of SiO$_2$/SiO$_2$, after etching treatment, is all the quartz fibers were left, which are the same regardless of the etching depth. However, it is worth noting that the CA of AgCuTi brazing alloy on the surface of E-SiO$_2$/SiO$_2$ decreased with the etching depth increasing, as shown in Fig. 8. In order to illustrate the wetting process, a concept physical model was established, as shown in Fig. 10. After etching treatment, the fused silica was consumed and quartz fibers were left in the transition zone, which contributed to the brazing alloy infiltrating into E-SiO$_2$/SiO$_2$. Furthermore, the width of the transition zone increased with the etching depth increasing, and then less and less brazing alloy was left on the surface of E-SiO$_2$/SiO$_2$, as shown in Fig. 10. Thus, the CA of E-SiO$_2$/SiO$_2$ decreased with etching depth increasing from the wetting experiments results. In fact, the CA of E-SiO$_2$/SiO$_2$ was constant. According to the above results, it is reasonable to infer that the space of the consumed silica sol can be filled up with brazing alloy, as long as it was enough.
Effect of surface structure of SiO$_2$/SiO$_2$ on the joint microstructure and property. In order to further investigate the relationship between the residual stress, the surface structure and the joint property in the brazed joints, the joining strength and the microstructure evolution was comparatively studied. The typical microstructure of 0 μm@E-SiO$_2$/SiO$_2$-Nb, 50 μm@E-SiO$_2$/SiO$_2$-Nb, 75 μm@E-SiO$_2$/SiO$_2$-Nb, 100 μm@E-SiO$_2$/SiO$_2$-Nb, 125 μm@E-SiO$_2$/SiO$_2$-Nb and 150 μm@E-SiO$_2$/SiO$_2$-Nb joints brazed by AgCuTi brazing alloy at 840°C for 10 min are shown in Fig. 11. It is obvious that the interfacial microstructure changed with the depth of brazing alloy infiltrating into SiO$_2$/SiO$_2$ increasing. As for 0 μm@E-SiO$_2$/SiO$_2$-Nb joint, continuous cracks can be observed in SiO$_2$/SiO$_2$ side near the brazing interface (see Fig. 11a). It may be because the poor wettability of SiO$_2$/SiO$_2$ and the high residual stress induced by the CTE mismatch between SiO$_2$/SiO$_2$ and Nb. So, the shear stress of the 0 μm@E-SiO$_2$/SiO$_2$-Nb joint was only 5 MPa (see Fig. 12). When SiO$_2$/SiO$_2$ after etching treatment, the brazed joint exhibited a 3D SiO$_2$/SiO$_2$-metal gradient transition zone and the width of the zone increased with the etching depth increasing (see Fig. 11b–d). Further, the brazing alloy was able to fill up the space between the quartz fibers and formed a sound metallurgical bonding with them, due to the great wettability of E-SiO$_2$/SiO$_2$. Furthermore, the “3D-pinning structure” can effectively reduce the residual stress in the joint, which decrease the continuous cracks and strengthen the joint. Thus, the shear strength of the joints increases from 5 MPa to 61.9 MPa with the etching depth increasing from 0 to 100 μm. However, the shear strength of the joints decreased with the etching depth increasing over 100 μm, because of the residual stress in the joints was increasing by the τ$_{xy}$ of the “3D-pinning structure”, according to the FEA results (see Fig. 12).
Figure 13. SEM images of the fracture surface in (a) 0μm@E-SiO₂f/SiO₂-Nb, (b) 50μm@E-SiO₂f/SiO₂-Nb, (c) 100μm@E-SiO₂f/SiO₂-Nb and (d) 150μm@E-SiO₂f/SiO₂-Nb joint.

Figure 14. XRD result patterns of fracture surface of (a) 0μm@E-SiO₂f/SiO₂-Nb, (b) 50μm@E-SiO₂f/SiO₂-Nb, (c) 100μm@E-SiO₂f/SiO₂-Nb, and (d) 150μm@E-SiO₂f/SiO₂-Nb joint.
In order to further study the effect of the transition zone on the obtained joints, the fracture analysis was conducted. Figure 13 shows the fracture surface of joints after shear tests. It can be seen that only remained broken quartz fibers were left in the fracture of SiO₂f/SiO₂-Nb joint, as shown in Fig. 13a, which may be due to the cracks at SiO₂f/SiO₂ side caused by the high CTE mismatch. By contrast, a posture of the same type of fracture morphology for the E-SiO₂f/SiO₂-Nb joints can be observed in Fig. 13b–d. The brazing alloy infiltrated into E-SiO₂f/SiO₂ can be observed in fractures. In addition, XRD was used to confirm the reaction products at the fracture surface. As shown in Fig. 14a, it can be seen that the fracture of E-SiO₂f/SiO₂-Nb joint was only composed of amorphous silicon dioxide. Correspondingly, amorphous silicon dioxide, TiSi₂, Ag(s,s) and Cu(s,s) were the reaction phases on the SiO₂f/SiO₂ composite fracture side for the fractures of 50μm@E-SiO₂f/SiO₂-Nb, 100μm@E-SiO₂f/SiO₂-Nb and 150μm@E-SiO₂f/SiO₂-Nb joints (see Fig. 14b–d). According to the XRD patterns, it can be inferred that as for SiO₂f/SiO₂-Nb joint, fracture occurred along the brazing interface on the SiO₂f/SiO₂ composite side. It may be due to the residual stress constraining around the SiO₂f/SiO₂-AgCuTi interface. Moreover, for 50μm@E-SiO₂f/SiO₂-Nb, 100μm@E-SiO₂f/SiO₂-Nb and 150μm@E-SiO₂f/SiO₂-Nb joint, fracture occurred in the 3D SiO₂f/SiO₂ metal gradient transition zone. Although the morphologies of the three kinds of fractures were almost the same, the mechanical properties of the joints were different, which may be owing to the residual stress concentrated in that zone.

Based on the above results, it is suggested that the FEA results can serve as a guide for the brazing test and theoretical basis for the distribution of residual stress in the brazed joints. In addition, the results show clearly that the etching treatment plays a key role in two major aspects on active brazing SiO₂f/SiO₂ and Nb. On the one hand, it can effectively improve the wettabiliy of SiO₂f/SiO₂, which is a precondition of successfully brazing SiO₂f/SiO₂ and Nb. On the other hand, it can induce the brazing alloy infiltrating into E-SiO₂f/SiO₂, which increases the bonded area, reduces the residual stress and enhances the mechanical properties. Therefore, regulating the surface structure is an easy and effective method to reduce the residual stress, which can strength the joints. As well the FEA and the brazing test results both reveal that the properties of 100μm@SiO₂f/SiO₂-Nb joint is the best among the obtained brazed joints with different surface structures.

Conclusions

In this paper, the optimized depth of brazing alloy infiltrating into SiO₂f/SiO₂ was achieved by combining FEA with experiments. After etching treatment, the fused silica with poor wettabiliy has been consumed while the quartz fibers with good wettabiliy were left, thus the wettabiliy of E-SiO₂f/SiO₂ was improved. The good wettability of SiO₂f/SiO₂ played an important role in obtaining a high-quality joint. Moreover, "3D-pinning structure" formed in E-SiO₂f/SiO₂-Nb joints, which can reduce the residual stress in the joint by form the sound gradient transition of CTE. However, the residual stress rather reduced with the etching depth increasing over appropriate size, due to the τy introduced by the "3D-pinning structure". A relationship between the residual stress, the surface morphology and the joint property in the brazed joints was demonstrated. As well the FEA and the brazing test results both realized the high-quality joint of E-SiO₂f/SiO₂-Nb joint and the shear strength of the joint reached 61.9MPa, which was approximately 12 times than that of SiO₂f/SiO₂-Nb joint.

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Conclusions

In this paper, the optimized depth of brazing alloy infiltrating into SiO₂f/SiO₂ was achieved by combining FEA with experiments. After etching treatment, the fused silica with poor wettabiliy has been consumed while the quartz fibers with good wettabiliy were left, thus the wettabiliy of E-SiO₂f/SiO₂ was improved. The good wettability of SiO₂f/SiO₂ played an important role in obtaining a high-quality joint. Moreover, "3D-pinning structure" formed in E-SiO₂f/SiO₂-Nb joints, which can reduce the residual stress in the joint by form the sound gradient transition of CTE. However, the residual stress rather reduced with the etching depth increasing over appropriate size, due to the τy introduced by the "3D-pinning structure". A relationship between the residual stress, the surface morphology and the joint property in the brazed joints was demonstrated. As well the FEA and the brazing test results both realized the high-quality joint of E-SiO₂f/SiO₂-Nb joint and the shear strength of the joint reached 61.9MPa, which was approximately 12 times than that of SiO₂f/SiO₂-Nb joint.

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Author Contributions

Jun Lei Qi and Qiang Ma conceived and designed the experiments. Jin Ba performed the experiments. Jing Huang analyzed the data. Lai Shan Yang prepared Figures 4–5. Qiang Ma performed the data analyses and wrote the main manuscript. Ju Lei Qi, Ji Cai Feng and Zhuo Ran Li contributed reagents/materials/analysis tools.

Additional Information

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