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Radiation effects on the mechanical properties of the material for the BESIII beam pipe supporting flange

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ABSTRACT

The supporting flanges were designed and made to support the Beijing Spectrometer (BESIII) beam pipe in Beijing Electron Positron Collider (BEPCII). They will be irradiated to neutron and gamma in their 10 years design life in BESIII. As one kind of excellent insulating materials, G10 epoxy glass cloth-laminated sheets (G10) was selected as the candidate material, and some experiments have to be performed to obtain information on the changes of mechanical properties after irradiation. According to the requirements of BESIII experiment, samples were irradiated by a ^{60}Co-source up to 10^4 Gy and in a reactor to a slow neutron fluence of $4.1\times10^{18}\,\mathrm{m^{-2}}$ ($E=0.625$ eV) sequently. The results show that the shear strength declines from 79.8 to 70.7 MPa. Since the established standards for tensile strength measurements involve sample sizes, which are far too large for the existing irradiation facilities, small size samples were designed to investigate the influence of the sample geometry on the tensile strength, based on which the tensile strength of G10 was deduced to decrease from 324.8 to 317.1 MPa after the same irradiation. The results indicate that G10 can well meet the requirements of the supporting flanges. Moreover, the shear and tensile fractures were observed by SEM to explain the experiment data. Now the supporting flanges made of G10 has been used in BEPCII supporting the BESIII beam pipe.

1. Introduction

A new detector, called Beijing Spectrometer (BESIII [1]), is under construction for the Beijing Electron Positron Collider (BEPCII [2]) at a center of mass energy of 1–2 GeV with a design luminosity of $1\times10^{33}\,\mathrm{cm^{-2}\,s^{-1}}$, with design life 10 years [3,4]. The BESIII beam pipe is located in the center of the BESIII and the electron and positron collide at the central region of the BESIII beam pipe, which is supported by two supporting flanges as shown in Fig. 1. The supporting flanges will be subjected to γ and neutron radiation over their 10 years lifetime in BESIII and the γ dose is 10^4 Gy and the neutron integral fluence is $4.1\times10^{18}\,\mathrm{m^{-2}}$. The BESIII experiment and the structure of the BESIII beam pipe require that the supporting flanges must be made of a material with excellent insulation and mechanical properties. Glass fiber-reinforced plastics (GFRPs) with excellent insulating property, consisting of boron-free glass fiber and matrix materials (e.g. multifunctional epoxy resins) are chosen as insulating material for the superconducting magnet coils of ITER. Accordingly, several kinds of GFRPs are researched by Bittner–Rohrhofer and Humer et al. The results show that GFRPs' mechanical properties, including tensile strength, short-beam and inter-laminar shear strength, etc., decline in varying degrees after the combined γ and neutron irradiation, even some material's strength after irradiation is unacceptable for ITER [5–11]. But the irradiation absorbed doses in those papers are completely different from that in BESIII. So, according to the BESIII work condition, some research on GFRPs must be done to guarantee the safety of the BESIII beam pipe. As one kind of GFRPs, G10 epoxy glass cloth-laminated sheets (G10) is chosen as the candidate material for the BESIII beam pipe supporting flanges. We undertook experiments to study its mechanical properties after irradiation. Aging phenomenon will take place in GFRPs without finishing agent in the air. The strength will decrease to 60% of its initial data after half of a year or 1 year, and then remains constant. As for as the GFRPs with finishing agent, significant decrease of its strength cannot be found after exposed to the air for 1 year [12]. The G10 in the experiment is dealt with finishing agent and has been exposed to the air for more than 1 year, so the aging phenomenon does not need to be considered in this paper. In different radiation environments, the experiment data of the inter-laminar shear strength of CTD-112, one kind of GFRPs show a similar trend [6]. Therefore, the influence of the dose rate on the mechanical strength of GFRPs is neglected and

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the irradiation experiment in the paper is done at a high dose rate to save time because 10 years is too long if the radiation condition in BESIII is simulated. The structure of the supporting flanges requires that the short-beam shear strength and tensile strength of G10 must be higher than 45 and 113 MPa, respectively. So the shear and tensile properties are researched in this paper.

2. Experiment

2.1. Material

The G10 in the experiment is manufactured by Guangzhou Taihe copper clad laminate factory, China. The reinforced fiber is S glass clothes (alkali content $< 0.5\%$) which is weaved in 7628 type. The rein matrix is E-20 epoxy resin, the curing agent is dicyandiamide, and the accelerator is 2 methylimidazole.

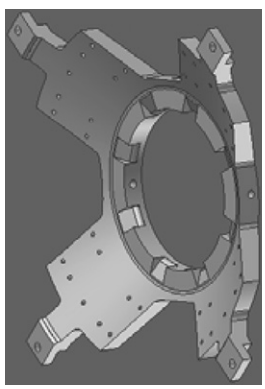

Fig. 1. Structure sketch of the supporting flange.

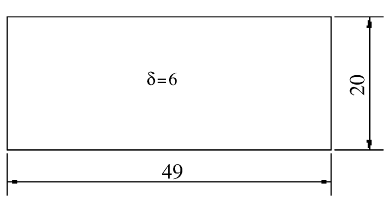

Fig. 2. Sample dimensions (mm) for the shear test.

2.2. Experimental samples

The samples for the shear test were prepared according to ASTM D 2344 and "Test method for the punch-type of strength of glass fiber-reinforced plastics GB/T 1450.2-2005". G10 was cut along the glass fiber and the samples were 40 mm long, 20 mm wide and 6 mm thick as shown in Fig. 2.

According to "Fiber-reinforced plastics composites determination of tensile properties GB/T 1447-2005", the tensile samples were 180 mm in length as shown in Fig. 3(a), which were considerably larger than the limited space in existing irradiation facilities, whose maximum length was 49 mm. So the small size samples were made, which was 49 mm long, which is 27.2% of the standard sample length as shown in Fig. 3(b).

2.3. Irradiation conditions

The γ irradiation was executed in air in the Academy of Military Medical Sciences, China. The samples were irradiated by a ^{60}Co-source up to 10^4 Gy with a γ-dose rate of 64 Gy h^{-1}. Then the samples were placed in a reactor of China Institute of Atomic Energy to a slow neutron fluence of 4.1×10^{18} m^{-2} ($E = 0.625$ eV) with the fluence density of 2×10^{17} m^{-2} s^{-1}.

2.4. Test procedures

The shear and tensile tests were done at room temperature. At least 5 samples were prepared for every type of test to guarantee 5 effective data. In the shear test, the loading direction was perpendicular to the G10 layer with one shear plane referencing to GB 1450.2-2005. M-100A electronic universal testing machine was used in the shear test. The tensile test was executed according to GB/T1447-2005 in RG300A electronic universal testing machine. Both the machines were manufactured by Shenzhen Reger Instruments Company and both the loading speeds were 2 mm min^{-1}.

The shear and tensile fractures were examined by Cambridge S-360 Scanning Electron Microscopy (SEM) to analyze the variation of the internal structure of G10 before and after irradiation.

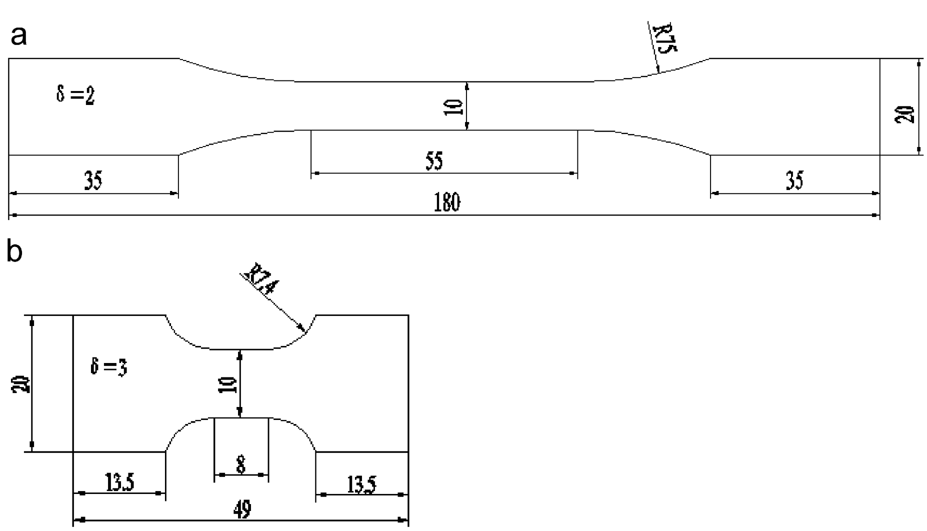

Fig. 3. Sample dimensions (mm) for the tensile test: (a) Standard sample and (b) small sample.

Table 1
Result of shear tests before and after irradiation

Irradiation dose	Cross-sectional area (mm^2)	Failure load (kN)	Shear strength (MPa)	Average shear strength (MPa)	Deviation (MPa)	Change rate
0	19.42 × 6.52	10.212	80.7	79.8	+0.9	–
	20.04 × 6.20	10.349	83.3		+3.5	
	20.00 × 6.52	10.118	77.6		−2.2	
	19.88 × 5.88	9.024	77.2		−2.6	
	20.00 × 6.50	10.408	80.1		+0.3	
γ: 10^4 Gy	19.92 × 6.00	8.875	74.3	75.1	−0.8	−5.9%
	20.10 × 6.00	8.960	74.3		−0.8	
	20.00 × 6.00	9.227	76.9		+1.8	
	19.88 × 6.38	9.535	75.2		+0.1	
	20.00 × 6.10	9.121	74.8		−0.3	
γ: 10^4 Gy neutron: $4.1 \times 10^{18}\,m^{-2}$	19.98 × 5.96	8.328	69.9	70.7	−0.8	−11.4%
	20.18 × 6.00	8.766	72.4		+1.7	
	20.04 × 6.08	8.792	72.2		+1.5	
	20.08 × 6.08	8.354	68.4		−2.3	
	20.04 × 6.08	8.600	70.6		−0.1	

3. Results and discussion

3.1. Shear property

In the shear test, failure load and dimensions were measured directly and the shear strength equaled to failure load divided by cross-sectional area. The results are listed in Table 1 and an overview is given in Fig. 4.

The shear strength of G10 is 79.8 MPa in the unirradiated state. After irradiation to 10^4 Gy γ and $4.1 \times 10^{18}\,m^{-2}$ neutron sequently, the shear strength degrades to 75.1 and 70.7 MPa, respectively. There are 5.9% and 11.4% proportional reductions.

The fracture sections of the shear samples are shown in Fig. 5. Comparing the three figures, it can be seen that the epoxy resin adhered on the glass fiber before irradiation is less than that after irradiation. No evident changes are found on the surface of the glass fiber after irradiation. But the destruction states of the epoxy resin vary before and after irradiation. The epoxy resin is a large sheet before irradiation (Fig. 5(a)), while that is small debris after irradiation (Fig. 5(b) and (c)).

An analysis of the corresponding load–displacement curve (Fig. 6) shows that the rate of load and displacement in Fig. 6(a) is larger than that in Fig. 6(b) and (c). That is to say that after irradiation to γ and neutron shear load reaches the maximum data until it precedes the longer displacement.

These results originated from variation of the internal structure of G10. The details can be described from three factors influencing the mechanical properties.

Firstly, the bonding strength between glass fiber and epoxy resin: the matrix, epoxy resin is the dispersion medium in G10 and the reinforced body, glass fiber is the dispersion phase. There is another phase in G10, which is the interface of epoxy resin and glass fiber. As multi-component material, G10 has some extra properties which do not exist in each one of the component. For instance, the strength of G10 is higher than that of epoxy resin cast and less than that of glass fiber. If the epoxy resin adheres to the glass fiber fully, the glass fiber can play its full reinforcing role and the strength of G10 will be stronger. On the contrary, its strength will be weaker [12]. In the experiments, the epoxy resin on the fracture glass fiber after irradiation is less than that before irradiation, which indicates that the bonding effect between glass fiber and epoxy resin is damaged by irradiation and the interfacial strength decline. The shear load cannot spread quickly from epoxy resin to glass fiber. So the reinforcing role of glass fiber is weakened and strength of G10 is affected, in good agreement with the load–displacement curve in Fig. 6.

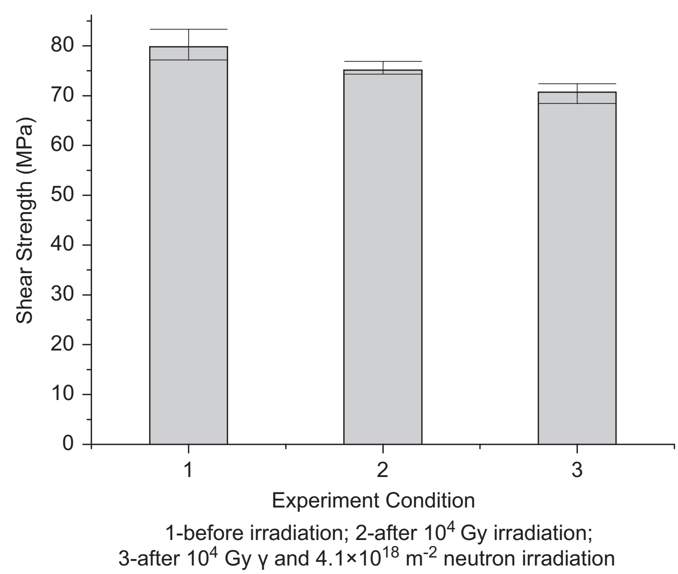

Fig. 4. Shear strength before and after irradiation.

Secondly, the strength of epoxy resin: crosslinking and degradation reaction occur simultaneously in high molecular polymeric material on the effect of irradiation. The ultimate reaction result depends on which reaction predominates. New crosslinking polymer generates when crosslinking reaction predominates over degradation reaction. Conversely, when degradation reaction predominates over crosslinking reaction, low molecular polymeric material is generated and some properties of the high molecular polymeric material disappear [13]. The obvious epoxy resin debris after irradiation indicates that the degradation reaction predominates over the crosslinking reaction in the irradiation process and the intermolecular force among the epoxy resin decreases. Therefore the epoxy resin is prone to damage and its shear strength to decrease. This is another factor that affects the strength of G10.

Lastly, the strength of glass fiber. Compared with any other fiber, glass fiber has the highest tensile strength, whose

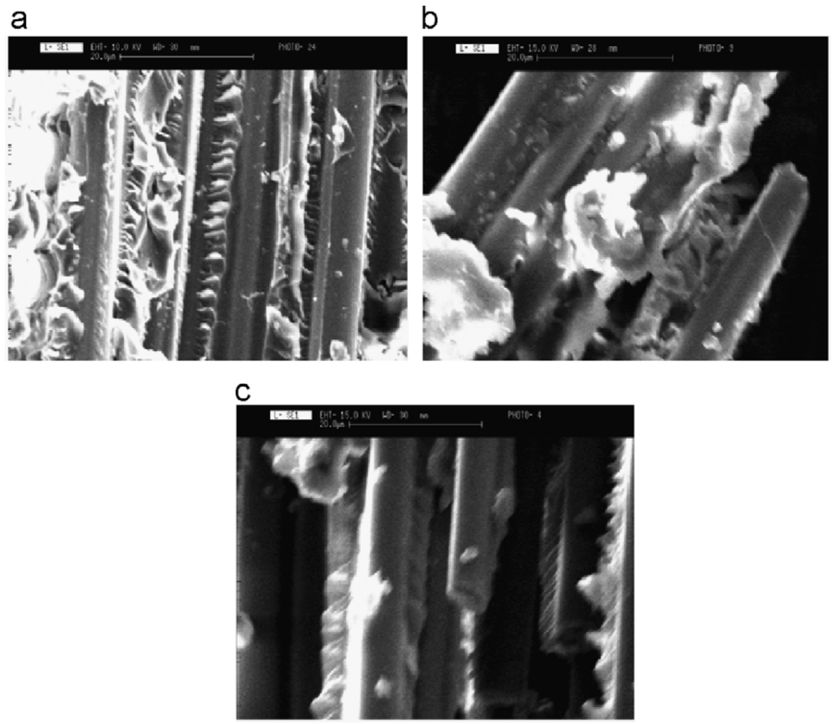

Fig. 5. Fracture sections of the shear samples: (a) before irradiation 2000×, (b) after 10^4 Gy γ irradiation 2000× and (c) after 10^4 Gy γ and $4.1\times10^{18}\,m^{-2}$ neutron irradiation 2000×.

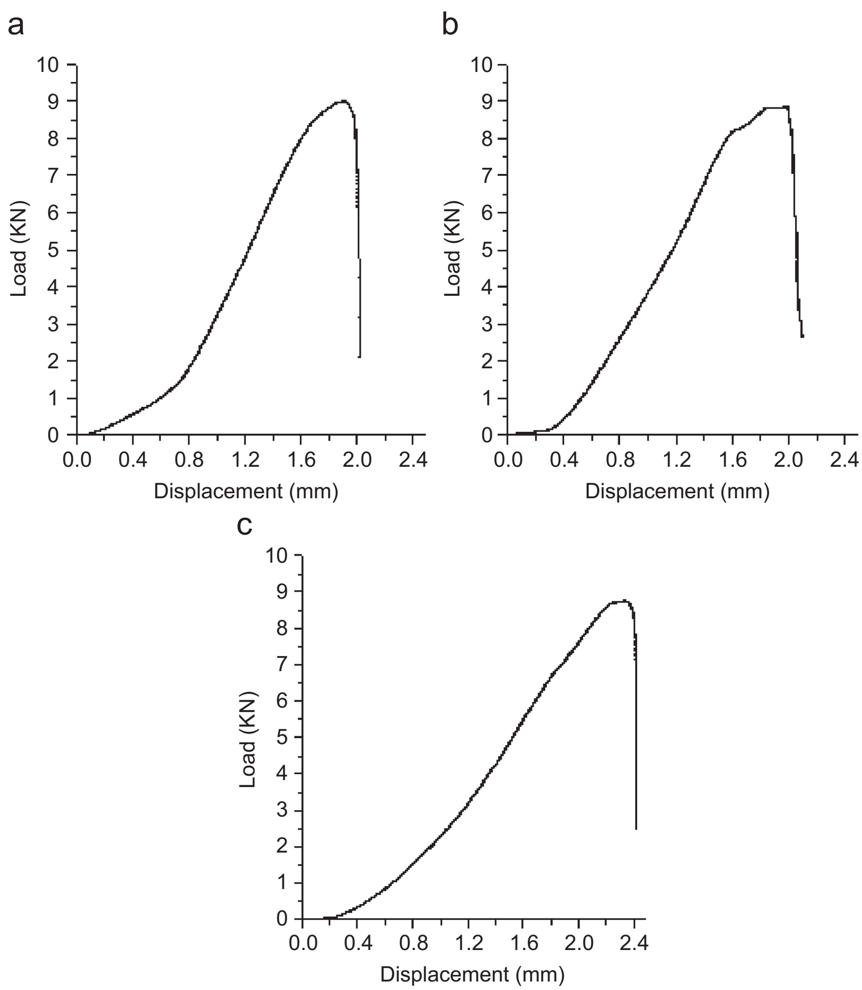

Fig. 6. Load–displacement curves for the shear test: (a) before irradiation, (b) after 10^4 Gy γ irradiation and (c) after 10^4 Gy γ and $4.1\times10^{18}\,m^{-2}$ neutron irradiation.

Table 2
Result of the standard and small samples for tensile tests before irradiation

Sample	Cross-sectional area (mm^2)	Failure load (kN)	Tensile strength (MPa)	Average tensile strength (MPa)	Deviation (MPa)	Change rate
Standard samples	9.92 × 1.84	5.711	312.9	324.8	−11.9	–
	10.00 × 1.86	6.251	336.1		11.3	
	9.92 × 1.84	6.209	340.2		15.4	
	10.00 × 1.84	5.840	317.4		−7.4	
	10.24 × 1.88	5.976	317.2		−7.6	
Small samples	9.82 × 3.14	5.620	182.3	160.3	22.0	−50.7%
	9.68 × 3.22	4.505	144.5		−15.8	
	9.42 × 3.12	4.680	159.2		−1.1	
	9.64 × 3.18	4.928	160.8		0.5	
	9.34 × 3.14	4.530	154.5		−5.8	

theoretical tensile strength, depending on the intermolecular force, is high up to 2000–12,000 MPa. Glass fiber is the primary stress body in G10 to reinforce epoxy resin and affect mostly the strength of G10. But there are many or few, large or small micro-cracks inevitably in glass fiber reducing its strength and affecting G10's strength. So the micro-cracks become the basic factors affecting G10's strength. No obvious changes occur on the surface of the glass fiber after irradiation, which demonstrates that the strength of glass fiber will not change remarkably and the strength of G10 will not vary greatly accordingly.

3.2. Tensile property

3.2.1. Influence of samples size on tensile property

In the case of the tensile behavior, all experiments should be done on the standard samples. Because of the limited space in existing irradiation facilities, smaller samples were prepared for the tensile property experiment. Different size samples consequentially lead to different experiment results, so the influence of the sample geometry on the tensile behavior must be studied. Similar problem occurs in ITER experiments. In Rosenkranz et al. [14] research, the ultimate tensile strength increase 10% and 20%, respectively, when the sample size reduces by 60% and 30%, and their smallest length is 70 mm. In this paper, the maximum length of the small size tensile samples was designed as 49 mm according to the existing irradiation facilities. Failure load and dimensions were measured directly and the shear strength equaled is defined as failure load divided by cross-sectional area. Table 2 gives the results for the influence of the sample sizes before irradiation and Fig. 7 gives the overview.

The results for the sample sizes investigated in our study show that before irradiation the tensile strength of standard sample is 324.8 MPa, while that of small samples is 160.3 MPa a decrease of 50.7%. The latter is 49.3% of the former. The reason can be described as follows. The holding length of the small sample is only 13.5 mm, which is less than that of the standard sample, 36 mm. So the holding force is heightened to avoid the small samples slipping from the holder, which results in the small sample breaking on the held position and the tensile strengths measured from small samples are much less than that from standard samples.

3.2.2. Influence of irradiation on tensile property

The tensile property of the small samples after irradiation to 10^4 Gy γ and $4.1\times10^{18}\,m^{-2}$ neutron were measured and the results are listed in Table 3 and an overview is given in Fig. 7.

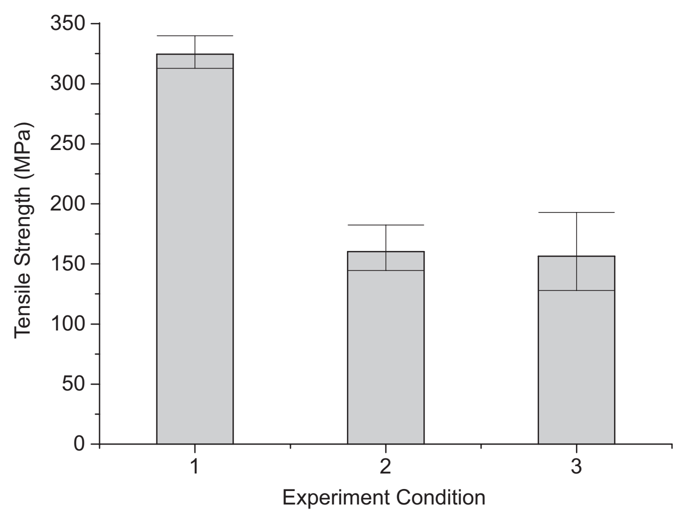

Fig. 7. Tensile strength before and after irradiation.

After irradiation to 10^4 Gy γ and $4.1\times10^{18}\,m^{-2}$ neutron sequently, the tensile strength of small samples is 156.5 MPa, a decrease of 2.4% compared to that of unirradiation state. Moreover, tensile fractures of small samples were examined by SEM as shown in Fig. 8.

Similar to Fig. 5, it can be seen from Fig. 8(a) and (b) that the epoxy resin before irradiation is large but small after irradiation. This illuminated the degradation reaction predominates over the crosslinking reaction in epoxy resin in the irradiation process and hence its tensile strength decreases. Therefore, the tensile strength of G10 decreases. It can also be seen that the surfaces of the glass fibers before and after irradiation are both smooth and obvious cracks cannot be found. Both the cross-sections of glass fibers in Fig. 8(c) and (d) are even and almost round. It is certain that as the reinforcing material, the glass fibers are not damaged by irradiation, so the strength of glass fiber and G10 will not change largely. But it can be found that after irradiation the glass fibers are pulled from the epoxy resin (Fig. 8(d)), which is not obvious before irradiation (Fig. 8(c)). That shows that the bonding strength between glass fiber and epoxy resin weaken after

Table 3
Result of the tensile property of the small sample after irradiation

Sample	Cross-sectional area (mm^2)	Failure load (KN)	Tensile strength (MPa)	Average tensile strength (MPa)	Deviation (MPa)	Change rate (compared with unirradiation data)
Small samples after irradiation	9.74 × 3.34	4.730	145.4	156.5	−11.1	−2.4%
	9.76 × 3.26	6.137	192.9		36.4	
	9.54 × 3.30	4.029	128.0		−28.5	
	9.76 × 3.24	5.018	158.7		2.2	
	9.70 × 3.12	4.766	157.5		1.0	

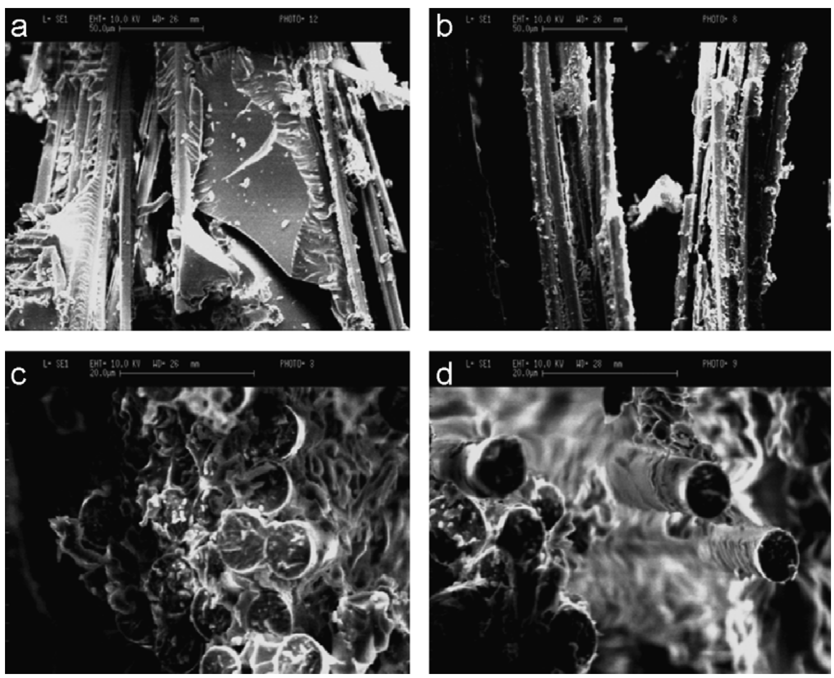

Fig. 8. Fracture sections of the small tensile samples: (a) before irradiation (wrap-wise) 500 × , (b) after irradiation (wrap-wise) 500 × , (c) before irradiation (latitudinal) 2000 × and (d) after irradiation (latitudinal) 2000 × .

irradiation, which does not contradict the previous result and lead to a decline of the tensile strength of G10.

Based on the experimental data and SEM pictures, we can presume that the change tendency of the small sample is suitable for the standard sample. So, it is inferred that the tensile strength of the standard sample will decrease 2.4% after irradiation, i.e. from 324.8 to 317.0 MPa.

4. Conclusions

After irradiation to γ dose of 10^4 Gy and neutron fluence of $4.1 \times 10^{18}\,m^{-2}$, mechanical properties of G10 changed much. Some conclusions can be drawn:

(1) It can be seen from the SEM pictures that the bonding effect between epoxy resins and glass fiber is damaged. The degradation reaction predominates over the crosslinking reaction in the irradiation process. Obvious damage on the glass fiber can not be found after irradiation.
(2) After irradiation to γ and neutron sequently, the shear strength declines 5.9% and 11.4%, to 75.1 and 70.7 MPa, respectively, from 79.8 MPa. This still meets the requirement, 45 MPa.
(3) After irradiation, the tensile strength of the small sample declines from 160.3 to 156.5 MPa, reduction proportion 2.4%. Based on the experimental data and SEM pictures, it can be assumed that the tensile strength of the standard sample will decrease from 324.8 to 317.0 MPa, which are higher than the requirement, 113 MPa.

In summary, as one kind of insulating material, G10's mechanical properties meet the requirements of the supporting flanges for the BESIII beam pipe on the irradiation conditions. Therefore, G10 is chosen as the material of the supporting flanges. Now the supporting flanges have been made and used in BEPCII.

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