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The design, fabrication and safety evaluation of a novel spent fuel storage basket material

Peng Wang[a,b], Xiaobin Tang[a,b,*], Feida Chen[a,b], Da Chen[a,b]

[a] Department of Nuclear Science and Engineering, Nanjing University of Aeronautics and Astronautics, Nanjing 210016, China
[b] Jiangsu Key Laboratory of Nuclear Energy Equipment Materials Engineering, China

HIGHLIGHTS

- Neutron absorption performance of the novel neutron absorbing material was improved by adding three different kinds of neutron absorbers (LiF, Sm_2O_3, and Gd_2O_3).
- The percentage of the three kinds of neutron absorbers was optimised by Monte Carlo method.
- Carbon fibre and polyimide were used to enhance its mechanical behaviour and thermal behaviour.
- The radiation effect of the neutron absorbing material had been studied under Co-60 irradiation, and its irradiation-resistance performance was evaluated.

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ABSTRACT

A novel LiF, Sm_2O_3, Gd_2O_3/carbon fibre/polyimide material was designed in order to improve the neutron absorbing performance of the spent fuel storage basket in this paper. The volume fraction of three kinds of neutron absorbers (LiF, $Sm_2O_3$and Gd_2O_3) in polyimide was optimised by Monte Carlo method. Calculation results showed that the novel neutron-absorbing material, in which the volume ratio of LiF, $Sm_2O_3$and Gd_2O_3 was 1:2:13, can achieve the best absorption capacity. Based on the calculated results, the basket material was fabricated by compression moulding, and its mechanical behaviour, thermal behaviour, and irradiation resistant behaviour were evaluated, respectively. The experimental results proved that the tensile strength of the novel neutron-absorbing material was between 195 and 346 MPa and the maximum service temperature was up to 300 °C. Gamma irradiation dose was limited to 160 kGy, the bending strength of the material kept increasing from 10 to 19 MPa.

1. Introduction

Spent fuel is defined as fuel that has been burned in a reactor. Compared with new fuel, the spent fuel has high radioactivity and can generate spallation neutrons by nuclear disintegration. Since the spallation neutrons may cause a nuclear criticality accident; using neutron absorbing materials, such as cadmium materials, boron plastics, B_4C/Al composites and boron steel, in its transportation and storage is an effective way of safely preventing criticality accidents. However, the aforementioned materials have their disadvantages: cadmium plate is a good absorber of thermal neutrons, but its absorption of epithermal neutrons is very weak (Abrefah et al., 2011). Boron plastic plays an important role as a neutron shielding material; for example, lead boron polyethylene and B_4C–PbO–$Al(OH)_3$–epoxy composite have extensive applications as neutron absorbing material (Ei-Sayed Abdo et al., 2003; Hu et al., 2008; Okuno, 2005; Sakurai et al., 2004). The tensile strength of B_4C–PbO–$Al(OH)_3$–epoxy composite is about 50 MPa and the maximum service temperature of lead boron polyethylene is 150 °C. However, compared with metal and metal matrix composites, there are still major gaps in mechanical and thermal performances. The manufacturing process of B_4C/Al composite is very complex (Halverson et al., 1989; Jung and Kang, 2004) and boron steel's boron content is insufficiently high for it to be applicable in this role (Bastürk et al., 2005; Zhang et al., 2013). Therefore, a novel neutron absorbing material needs to be researched

* Corresponding author. Present address: Department of Nuclear Science and Engineering, Nanjing University of Aeronautics and Astronautics, 29 Yudao St., Nanjing 210016, China. Tel.: +86 13601582233; fax: +86 02552112906 80407.
E-mail addresses: wp739130046@nuaa.edu.cn (P. Wang), tangxiaobin@nuaa.edu.cn (X. Tang), fdchen@nuaa.edu.cn (F. Chen), chenda@nuaa.edu.cn (D. Chen).

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to cope with the problem of neutron absorption from spent fuel.

With the purpose for improving the performance of neutron absorbing materials, this research studied a novel neutron shielding material for the use in spent fuel storage basket. Through adding different neutron absorbers, the basket material can absorb neutrons of different energy. A continuous carbon fibre reinforced polyimide resin was selected as the matrix for the new basket composite material to improve its heat resistance and mechanical properties. Because carbon fibre reinforced polyimide resin has high specific strength, high specific modulus, excellent temperature resistance, and excellent corrosion resistance; it is one type of high-performance material widely applied across various engineering applications. In addition, the neutron absorbing material can also be used in the spaceflight and for radiation protection.

2. Experimental methods

2.1. Calculation model

Compared with protons and electrons, neutrons have no charge and are strongly penetrability. The process of any neutron reaction with matter is divided into two steps: scattering and absorption. Generally, neutrons decrease their energy by scattering and their absorption by nucleus reduces its total number. The polymer is conceptually an excellent neutron-moderator and shielding material (*e.g.* polyimide resin). Substances with large absorption cross-section are chosen as neutron absorbers, *e.g.* ^{10}B, ^{6}Li, or ^{157}Gd. Traditional basket materials for spent fuel storage use boron carbide as a neutron absorber. Even though boron carbide is good neutron absorber, some rare earth elements have a larger neutron absorption cross-section (Cao et al., 2010). Therefore we chose the rare earth elements (*e.g.* Gd and Sm) for improving neutron absorptivity instead of boron.

A type of neutron absorber only absorbs neutrons with a specific range of energies, but the spent fuel's neutron energy range was too wide for a type of neutron absorber. A type of neutron absorber might not absorb all energy neutrons, so three different neutron absorbers were used in attempts to solve this problem (see Fig. 1(a)). In this work, the neutron energy spectrum of spent fuel assemblies with an initial uranium-235 enrichment of 4%, discharge burn-up of 45 GWD/MTU, and a 5-year cooling time was used (see Fig. 1(b)) (Zhang, 2010). The absorption behaviour of 10 mm thick neutron absorbing material was calculated to find the optimum volume fraction of three different neutron absorbers.

MCNP is particle transport simulation software and has a higher precision in radiation calculations (Ranft, 1967). Many shielding materials were designed by MCNP code (Khan et al., 2011; Miri-Hakimabad et al., 2007). In this work, MCNP-4C was used to design the novel neutron absorbing material.

In order to improve neutron absorption capability of the novel neutron absorbing material, the higher volume fraction of the overall neutron absorbers is better. However, the novel neutron absorbing material could not been formed if the volume fraction of the overall neutron absorbers was higher than 15%. Therefore the maximum volume fraction of the overall neutron absorbers was 15%.

A spherical shell model was established to determine the content for each neutron absorber. With the volume fraction of overall neutron absorbers being constant, the relative proportion of three absorbers was changed in calculations to obtain their neutron transmissivity. From the centre of each spherical shell to the outermost range; the point source was placed at the centre, followed by air and the neutron absorbing material. The outermost part of the model was the count surface and the F2 card (record of average surface flux) was used to assess the number of neutrons transmitted (see Fig. 2).

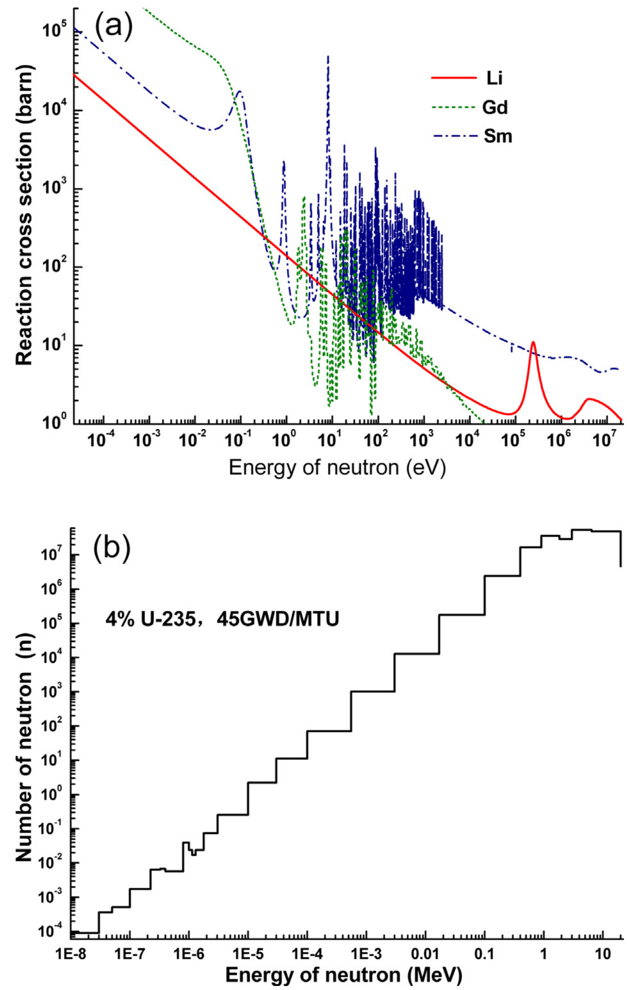

Fig. 1. (a) Neutron cross section of three kinds of neutron absorbers (LiF, Sm_2O_3, and Gd_2O_3). (b) Neutron spectrum of spent fuel (4 wt% ^{235}U, 45 GWD/MTU, cooling time 5 years).

2.2. Fabrication of polymer materials

A polyacrylonitrile (PAN)-based carbon fibre cloth (with a thickness of 0.3 mm) and a thermosetting polyimide resin (Model:

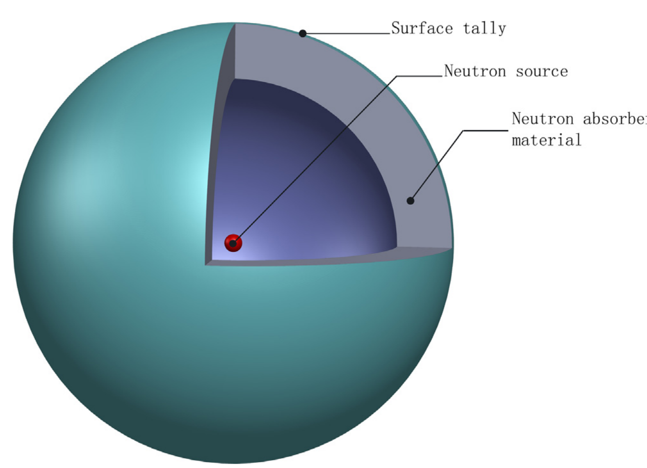

Fig. 2. The spherical shell model used for calculation.

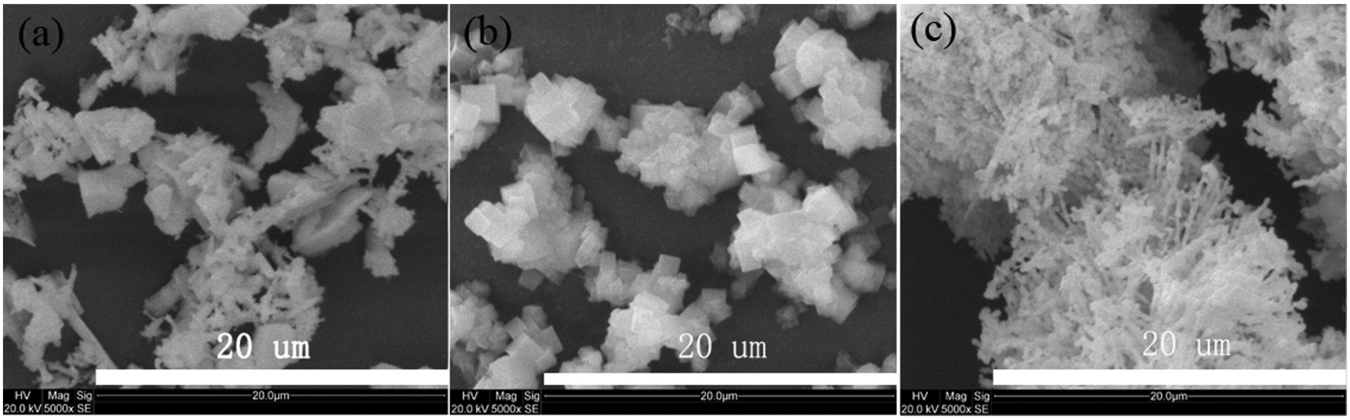

Fig. 3. The microstructures of three kinds of neutron absorbers (a) LiF; (b) Sm_2O_3; and (c) Gd_2O_3.

TY005-1) were selected as reinforcement and matrix for the neutron absorbing material, respectively. Three different kinds of powder (Gd_2O_3, Sm_2O_3, and LiF) with purities exceeding 99.5% were added as neutron absorbers. Because small particles could avoid sedimentation problem, these particles were sub-2 μm diameter (see Fig. 3). Since polyimide could dissolve in organic solvents, acetone was chosen as the diluting agent.

According to simulated results, the novel type of neutron absorbing material was manufactured. The polyimide resin was heated to 60 °C to decrease its viscosity. When the polyimide had sufficient fluidity, acetone was added to dilute it, and then we added neutron absorbing particles into the diluted resin and stirred the mixture for 30 min. As shown in Fig. 4, carbon fibre cloths were cut into 30 cm squares, then dip-treated in the resin mixture and laid-up one by one. Finally, the novel neutron absorbing material was formed by hot-pressing.

2.3. The coefficient of thermal conductivity test

To ensure the decay heat produced by the spent fuel could be removed in time, it was necessary to evaluate the thermal conductivity of the spent fuel storage basket material. The steady-state bi-substrate technique was required for measuring the thermal conductivity of the neutron absorbing materials (Tan et al., 2006). The thermal conductivity tester's sensitive face was divided into a heating surface and a radiating surface: the sensitive face was made of copper and had a diameter of 30 mm. In addition it was surrounded by polystyrene foamed plastics for thermal insulation. Three various thicknesses of disk were required for the measurement of thermal conductivity of the material. Three samples with various thicknesses, 0.90 ± 0.1 mm, 1.26 ± 0.1 mm, and 1.64 ± 0.1 mm, were chosen and made into wafers about 30 mm in diameter to assess the heat transfer performance of the neutron absorbing material. The heating surface's temperature was 70 °C for this test.

$$q = \lambda_{\text{eff}} \frac{\Delta T}{d_S} \tag{1}$$

$$q = h \Delta T_1 \tag{2}$$

$$q = \lambda_{\text{true}} \frac{\Delta T_2}{d_S} \tag{3}$$

where λ_{eff} is the effective thermal conductivity of each material, λ_{true} is the true thermal conductivity of each material, d_S is the sample thickness, ΔT_1 and ΔT_2, are the thermal drop across the surface and samples, and ΔT is the total thermal drop, $\Delta T/q$ was given by the test apparatus, and h is the heat conductance of surface.

The total temperature drop, ΔT is composed of drops across the sample and the surfaces.

$$\Delta T = \Delta T_2 + m \cdot T_1 \tag{4}$$

where m is the number of surfaces. By substituting Eqs. (1) and (2) into Eq. (4), and rearranging:

$$\frac{\Delta T}{q} = \frac{d_s}{\lambda_{\text{true}}} + \frac{m}{h} \tag{5}$$

The thermal conductivity was calculated using Eq. (5).

2.4. Thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC)

During spent fuel storage, because spent fuel generates great deal of heat, the storage environment temperature is relatively high. Hence the thermal properties of the neutron absorbing material should be evaluated. The heat resistance of this composite material was analysed by TGA–DSC coupling technology in air. The sample of mass 8.51 mg was placed in the differential scanning calorimetry apparatus, and a heating rate during TGA–DSC of 10 °C/min and a maximum temperature of 400 °C were used.

2.5. Mechanical test

Since the spent fuel storage basket plays a supporting role for spent fuel assemblies, the materials needs to have good mechanical properties. Mechanical testing was used for studying the effects of the number of carbon fibre sheets on the mechanical properties of the neutron absorbing material. The tensile properties of composites with different layers of carbon fibre sheets (1, 4, 6, 8, 15, and 40 layers) were tested and compared. The tensile displacement rate of 2 mm/min was maintained during the tests, and span distance of the grips was 115 mm. Based on measurement standards, the novel absorbing neutron material was processed to dumbbell specimens for tensile test. The maximum tension capacity of the tensile testing machine was 100 kN (considerably larger than the capacity of the absorbing neutron material).

2.6. Irradiation experiment

During spent fuel storage, and because spent fuel assemblies are highly radioactive, spent fuel storage baskets have to undergo significant irradiation. The mechanical properties of the material were affected by irradiation. To assess the influence of irradiation, 4×10^5 Ci from a ^{60}Co source was chosen for this irradiation experiment at a dose rate of 1 kGy/h. The samples were 8 ± 1 mm thick composite materials and the irradiation doses were 60 kGy,

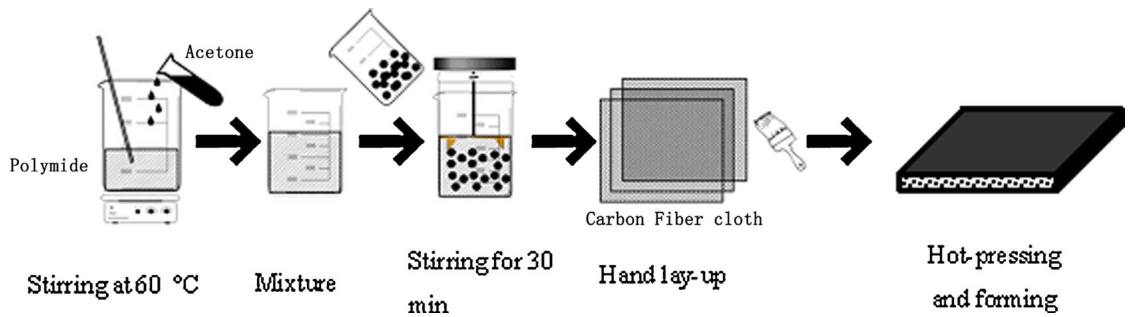

Fig. 4. The main steps for fabrication of spent fuel storage basket material.

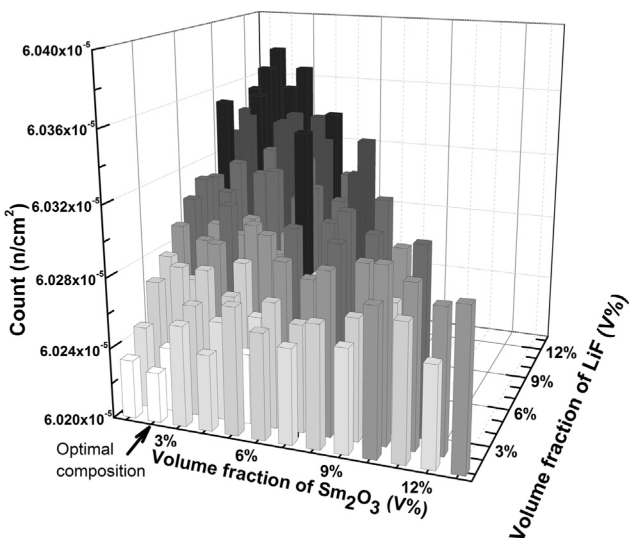

Fig. 5. Count statistics for different proportions of neutron absorber.

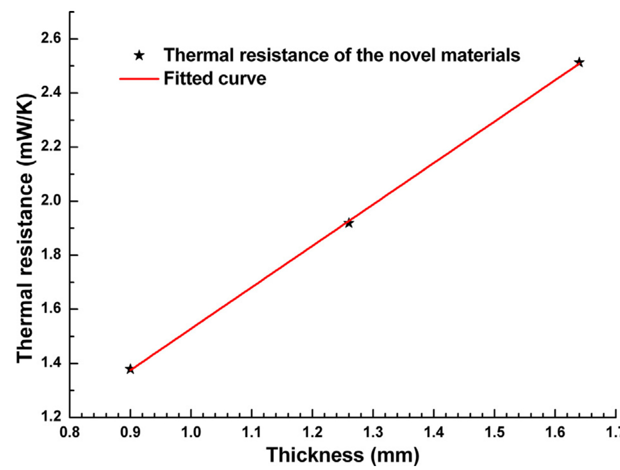

Fig. 6. Measured thermal conductivity data for the novel neutron-absorbing material.

90 kGy, 120 kGy, and 160 kGy. Since irradiation has more impact on the resin, the bending properties of this novel neutron absorbing material were analysed.

2.7. Fourier translation infrared spectroscopy (FTIR)

FTIR is a kind of effective way to analyse the chemical structure of materials. In order to study the structure stability of the novel neutron absorbing material in the process of irradiation, the FTIR was used to test the changes of functional groups after irradiation. To obtain the ideal results, block samples irradiated by different doses were grinded into powder. Spectral range was 400–3800 cm^{-1}, and scanning times is 32.

3. Results and discussion

The total volume fraction of three different neutron absorbers was 15% (*i.e.* V_1% (samarium oxide) + V_2% (gadolinium oxide) + V_3% (lithium fluoride) = 15%. We changed the volume ratios of three different neutron absorbers, and then recorded the number of neutrons that were not absorbed after passing through a 10 mm thick sample of the neutron absorbing material to draw Fig. 5.

As shown in Fig. 5, based on the comparison of results after normalisation, the neutron absorption performance of the material gradually degraded as the volume fraction of lithium fluoride increased for a certain volume fraction of samarium oxide. That meant that there was an upper limit to the lithium fluoride content, although it did have a good absorption effect for specific energy neutrons. As the volume fraction of samarium oxide increased, there was little difference in the neutron absorption performance of the material. But overall, Gd played a greater role in neutron absorption, and the Li and Sm had a supplement for Gd. In Fig. 5, when the volume fractions ratio of three different neutron absorbers was 1 LiF:2 Sm_2O_3:12 Gd_2O_3, the neutron absorbing material had the best neutron absorption performance.

Table 1 lists the results from tests on different neutron absorbers for the same base material in analogous calculations. The matrix of these materials was a polyimide resin enhanced by continuous carbon fibres with different neutron absorber types. Each neutron absorber could capture neutrons of different energies as an individual; however the combination with the given ratio gave the minimum neutron transmission. This meant that the method using a blend of different types of neutron absorber could most effectively enhance the overall neutron absorption capacity.

According to Eq. (5), plotting $\Delta T/q$ *versus* d_s, the slope and intercept were given by $1/\lambda_{\text{true}}$ and m/h, respectively. Both $1/\lambda_{\text{true}}$ and h can thus be determined from such a plot. As shown in Fig. 6, 0.9 mm, 1.26 mm, and 1.64 mm thick samples of neutron absorbing material were chosen for the experiment: the slope and intercept of the plot were 1.53 and 0, respectively. Because of the intimacy of contact between material surface and instrument, the heat conductance of the surface was infinite. The coefficient of thermal conductivity was 0.65 W/(m K).

The heat conductance of traditional boron plastic with 15 vol% neutron absorber is 0.5–0.7 W/(m K). Compared with metal, the thermal conductivity of resin matrix is poor relatively. For polyimide resin, the thermal conductivity is not higher than 0.35 W/(m K) (offered by the manufacturers). Powder and fibre contribute to improving thermal conductivity. According to the results

Table 1
Neutron absorption performance of different materials.

Neutron absorber	Volume fraction (*V*%)	Theoretical density (g/cm^3)	Thickness (mm)	Surface counts (*n*/cm^2)	Statistical errors (%)
B_4C	15%	1.7	10	6.02807×10^{-5}	0.04
LiF	15%	1.7	10	6.03750×10^{-5}	0.04
Sm_2O_3	15%	2.1	10	6.03620×10^{-5}	0.04
Gd_2O_3	15%	2.02	10	6.02613×10^{-5}	0.04
LiF, Sm_2O_3, Gd_2O_3	15%	2.00	10	6.02283×10^{-5}	0.04

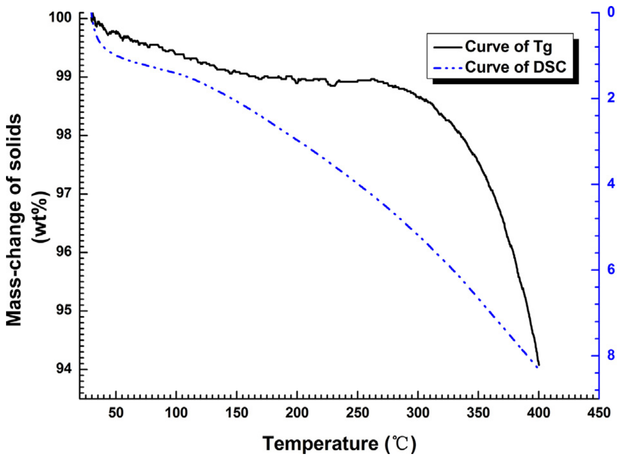

Fig. 7. The novel neutron absorbing material's TGA and DSC curves.

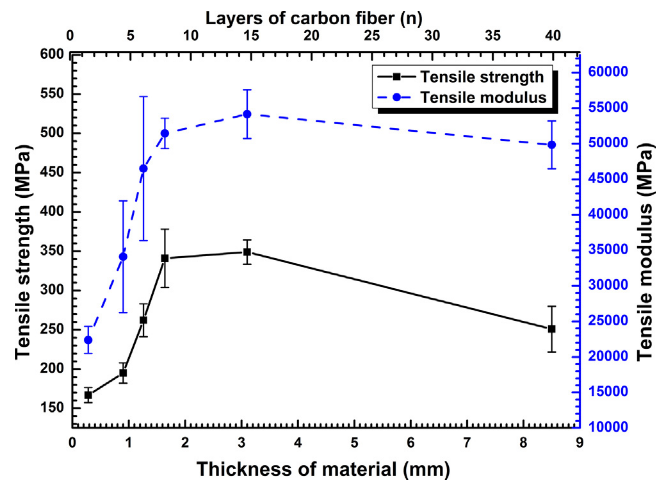

Fig. 8. The neutron absorbing material's tensile strength test data.

above, the novel neutron absorbing material can reach the level of traditional boron plastic in the vertical direction.

At high temperature, cross-linking, glass-rubber transition, and pyrolysis occur mainly in the polymer composite. As seen from Fig. 7, the DSC plot increased linearly with temperature. The DSC plot can show the process of cross-linking and glass-rubber transition. Cross-linking can form endothermic and exothermic peaks; however no obvious peaks were seen here. The neutron absorbing material had already completely cured. If glass-rubber transition occurred in resin materials, the DSC plot would have been S-shaped: as such, this neutron absorbing material was stable between 30 and 400 °C.

In Fig. 7, the TGA curve decreases rapidly starting from 300 °C; this could be attributed to the fact that the investigated polymeric composite is of high molecular weight and it could be starting to decompose at such high temperature environment. However, the material underwent no structural change. Therefore the neutron absorbing material's degradation temperature was as high as 300 °C.

As the matrix cannot withstand high temperature, the traditional boron plastics' maximum service temperature was about 150 °C. The new type of neutron absorbing material's the maximum service temperature was twice as high as the traditional boron plastics', so it had better properties of heat resistance.

In Fig. 8, the mechanical test experimental results showed that in the samples thicknesses range from 0.2 to 8.5 mm conditions, the modulus of the material was between 22.39 and 54.16 GPa, the ultimate tensile strengths distributed between 166.88 and 341.03 MPa. There were two main factors that influenced the tensile properties of the material: stress transfer in the resin, and interfacial defects. Stress transfer between the polyimide resin and the carbon fibre decreased the stress concentration which improved the material's tensile strength, but interfacial defects impaired the composite material's mechanical properties. Within the maximum distance of stress transfer, the tensile strength increased with the increased number of carbon fibre layers. However, the more carbon fibre layers, the more interfacial defects were present, once the thickness of the composite materials exceeded the maximum distance of stress transfer, the tensile strength and modulus decreased with the number of carbon fibre layers. In addition, the material was too thick, and there were stress concentrations appearing near the chucks of the tensile testing machine, thus the tensile strength decreased more rapidly.

The tensile strength of conventional aluminium matrix composites is about 200–300 MPa, and the tensile strength of boron plastic is about 20–50 MPa. The novel neutron absorbing material has more excellent tensile strength than traditional boron plastic. The novel neutron absorbing material has reached the level of conventional aluminium, and can meet the requirements for spent fuel storage and transportation purposes.

As shown in Fig. 9, the bending strength firstly increased and then remained steady with the increase of dose from zero to 160 kGy for the shielding materials with 40 lays carbon fibre cloth. Due to the cross-linking reaction occurring after exposure

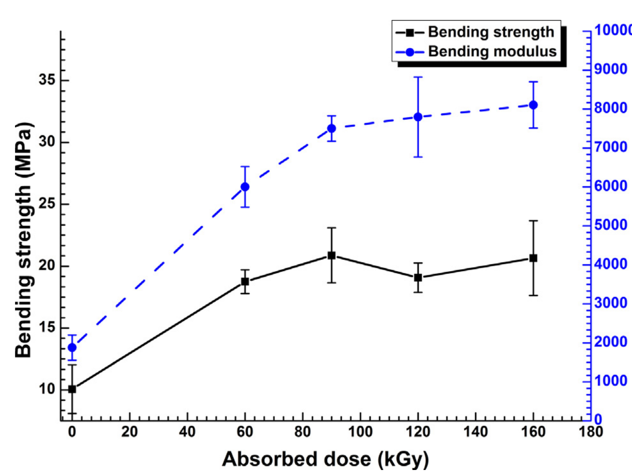

Fig. 9. Bending performance of the neutron absorbing material under different irradiation doses.

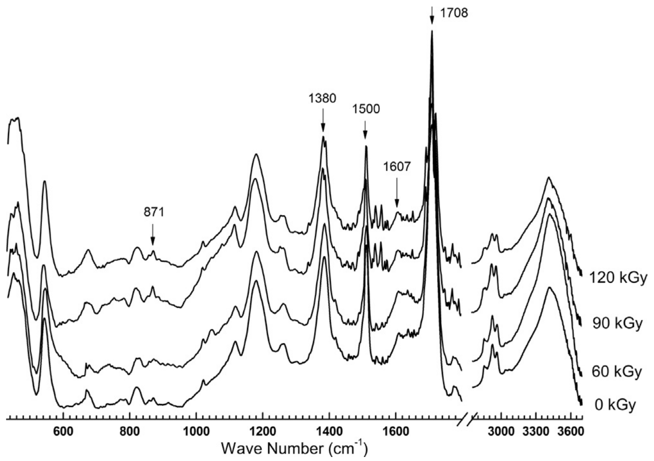

Fig. 10. The IR spectra for the selected neutron absorbing material before and after irradiation.

to nuclear radiation, the polymer structure was more stable. Therefore, under conditions of low dose irradiation, the flexural strength and flexure modulus of such composites had been greatly increased. However, with increasing irradiation dose, the number of chemical bonds whose bond energy was low became very small. High-dose irradiation reduced the rate of cross-linking, so the flexural strength and flexure modulus of these composites tended to be stable.

In order to find out the reason for the change in mechanical properties, alterations of the chemical structure were studied by Fourier transform infrared spectroscopy. Under different irradiation doses, the IR spectra of the neutron absorbing material were as shown in Fig. 10. At 1380 cm^{-1} there was an absorption peak attributed to the C–N bond in the polyimide: at 1500 cm^{-1} and 1708 cm^{-1} were stretching vibrational absorption peaks from the p-substituted benzene ring, and asymmetric carbonyl component of the pentatomic-imine ring (Diaham et al., 2011; Karamancheva et al., 1999). The material structure had not greatly changed albeit some details were different; *e.g.* the out-of-plane flexural vibration of the =C–H bond at 871 cm^{-1} gradually increased, the C=O symmetrical stretching vibrations located at 1607 cm^{-1} were increasingly apparent which proved that a few acid amide groups were generated at high irradiation doses. However, it did not affect the integrity of the material's structure, and the novel neutron absorbing material had satisfactory radiation-resistance performance facing irradiation up to the aforementioned dose.

4. Conclusion

In conclusion, we optimised the compositions of the novel neutron absorbing material through Monte Carlo simulation, and certified that samarium oxide and lithium fluoride could supplement for the absorption capacity of the gadolinium oxide. After the volume fraction of three kinds of neutron absorbers optimised, the novel neutron absorbing material was manufactured by hot-press method, and its properties had been tested. Test results confirmed that the novel neutron absorbing material had good mechanical properties, sufficient heat tolerance, satisfactory thermal conductivity, and enough irradiation resistance to work in the spent fuel storage environment. Its tensile strength was greater than 200 MPa, the highest temperature tolerance and coefficient of thermal conductivity were 300 °C and 0.65 W/(m K), respectively. The structure of the material was stable at irradiation doses of up to 160 kGy. Moreover, manufacturing technology of composite material has many characteristics, such as simple technology, saving energy and low cost. Therefore, the novel neutron absorbing material can be used to make spent fuel storage basket and spent fuel storage canister and has a good application potential for nuclear waste management.

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