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Research of the thermal stability of structure of resin anchoring material based on 3D CT

Su Xue-gui [a,b,*], Du Xian-jie [a], Yuan Hong-hu [a], Li Ben-kui [a]

[a] College of Mining Engineering, Taiyuan University of Technology, Taiyuan 030024, China
[b] State Key Laboratory for Geomechanics and Deep Underground Engineering, China University of Mining and Technology, Xuzhou, Jiangsu 221008, China

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ABSTRACT

As a polymer material, UPR anchoring material has pyrolysis at high temperature, which directly affect its mechanical stability. Using micro-CT scans and 3D image reconstruction to analysis pyrolysis characteristics and the pore structure evolution of UPR anchoring material at high temperatures, and the results showed that the thermal decomposition cause obvious changes in internal microstructure at high temperatures; the average gray attenuated 20.7%, pore groups number increased 47.5% and pore group size increased 201.5% between 350 °C and 500 °C; organic cemented body corresponding to attenuation coefficient ranges from 0.0163 to 0.0373, the inner pore increased significantly after organic pyrolysis, and connected with each other to form larger pores group; the pyrolysis critical temperature is 350 °C, pyrolysis affects significantly to the internal structure and the density of the material. Mechanical properties decayed significantly at high temperature (400 °C), compressive strength decreased by 95% and pull-out strength decreased by 68.3%.

1. Introduction

Based on different types of resin, resin anchoring material can be divided into unsaturated polyester resin (UPR), epoxy resin, polyurethane resin and so on. Because of its fast gelling speed, high early strength (compressive strength of 30 min under normal temperature can reach 50–60 MPa), low cost and widely adaptation, the UPR anchoring material was used in coal mine roadway supporting, building strengthening and other projects. The UPR anchoring material is a clay-like adhesive material mixed by UPR and $CaCO_3$ powder on the scale of 5:1, then it was mixed with an appropriate amount of accelerator. To package and divide the clay and curing agent into a roll using polyester film, then use the mechanical stirring method to mix the two components fully for bonding and reinforcement [1–4]. But as the coal mines and other underground engineering extend to 1000 m underground and below, geothermal and spontaneous combustion of coal seam are becoming more frequent, which lead to the temperature rising of surrounding rock of underground engineering [5–8], especially when the coal mine gas explosion happens or the buildings on the ground burst fire and the structure temperature reaches to 500–800 °C [9–12]. Therefore, there is a higher requirement on the mechanical stability of resin anchoring material at high temperature. UPR anchoring material has pyrolysis properties at high temperatures as an organic polymer composites, and the changes of the internal structure at high temperature will directly affect the mechanical properties of the material [13]. Therefore, to study the characteristics UPR anchoring material under high temperature (20–500 °C) and its dense internal structure evolution regularity based on the three-dimensional(3D) micro CT image is very important for the further research of thermal decomposition, and it provides scientific basis to the research of adapting to the underground engineering of deep and construction reinforced high temperature resistant anchoring material.

Application study on UPR composite material is relative to many fields. Many scholars have already done some research on the resin internal structure and low temperature heat properties. Kang Hongpu and Hu Bin have studied on the effect of the temperatures on the properties of UPR anchoring material by using pull-out test and numerical simulation study, the results showed that the temperature have great influence on the anchoring force, but the temperature scope of the researches are limited to 20–85 °C [14,15]; Fan Shiping has carried out a study on the feasibility of high temperature resistant UPR anchoring material (120 °C) by using TGA from the perspective of the introduction of a crosslinking group of good thermal stability to prove the feasibility of the production and the use of high temperature resistant UPR anchoring material [16]; Baskaran, Rajendran applied TEM, XRD and SEM to show that the mechanical and thermal properties of UPR/nano $CaCO_3$ composites

* Corresponding author at: College of Mining Engineering, Taiyuan University of Technology, Taiyuan 030024, China. Tel.: +86 351 6014501.
E-mail address: 666sxg@163.com (S. Xue-gui).

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are better than those of pure UPR [17]; Haitham Elbishart applied micro-CT to the study of the correlation between resin composites filler and their structure, and he came to the conclusion that filler size was strongly correlated to void percentage, but it had no effect on fracture toughness [18]; Eitetsu Choa came up with the application of micro-CT and sophisticated image analysis, which was a novel approach to investigate shrinkage mechanisms of dental composites. He achieved good results by assessing the polymerization of the composite resin through the micro-CT imaging and nano-indentation technique [19]. Although micro-CT has been recognized as a powerful tool for obtaining information on the microstructure of the adhesive [20], the research of pyrolysis process under high temperature (20–500 °C) and its internal microstructure of $CaCO_3$ powder/UPR composite anchoring material under 500 °C high temperature based on the 3D micro-CT is limited.

In this study, the researcher scanned the UPR anchoring material samples at different temperatures (20–500 °C) by high-precision test analysis system with micro-CT(μm level) and studied the pyrolysis characteristics and porosity evolution of UPR anchoring materials by applying 3D image reconstruction to verify the results by the experimental results of the high temperature mechanical properties of the composite material.

2. CT experimental procedure

2.1. Equipment and principle of CT experiment

The Micro-CT test equipment is high-precision micro-CT μCT225kVFCB test analysis systems [21], as shown in Fig. 1. Micro CT test analysis system of μCT225kVFCB high precision (μm level) type is mainly composed of a micro focus X-ray machine, the digital plane detector, high precision turntable and clamp, a machine seat, a horizontal movement device, acquisition analysis system and other structural parts. The minimum focus of the system is 3 μm, the focal length is 4.5 mm, the flat panel detector probe size is 0.194 mm, the amplification multiple is 1–400, the density resolution is ≦0.2%, and it can recognize pores of 0.5 μm. In order to obtain the evolution of the internal structure at different temperatures of the same part of material, the test used home-made atmosphere furnace heating, as shown in Fig. 2.

As shown in Fig. 3, the turntable is rotating at a uniform speed in the CT scanning process, X ray is scanning the longitudinal profile of the object, and each scan profile corresponds to a set of projection data. The essence of CT scan is that the strength of X ray is certain before it went through the object but it will change after it went through the object because of the density of the object

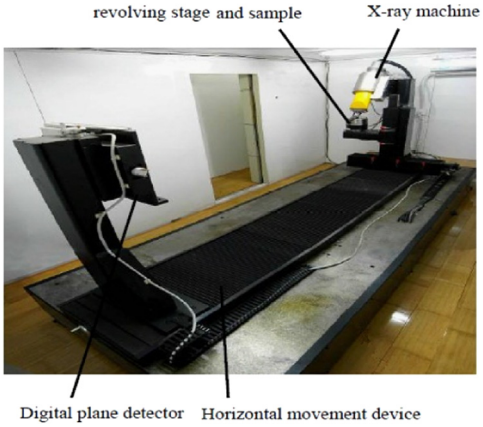

Fig. 1. Micro-CT system.

changes. The attenuation coefficient of each unit was obtained based on the relationship between the X-rays light intensity difference before and after the test piece penetration and the size and density of the specimen, and the the gray value of the test piece was converted to generate CT images at the end. CT image is the arrangement of pixels, and the size of the attenuation coefficient of each volume element represents the pixel value of the point, and the gray value of CT gray image is converted from the pixel value. So in the CT gray image, different colors presented material of different density. The greater the density of the material, the higher the brightness.

2.2. Preparation of CT test pieces

In order to get a more direct presentation of the UPR anchoring material's internal structural that changes with the temperature and to have a statistical comparison of data, the same specimen was performed to scan at different temperatures, and the same area was performed for 3D reconstruction. CT test specimen of resin anchor material,which has been blended, was made into the test block of 70.7 mm × 70.7 mm × 70.7 mm with a molding process of using the curing agent, of which the gel time was 10–15 min, and quickly mixing evenly with the resin cement and put them into the mold. Then calibrating it by mechanical vibration for 1 min, and released it from the mold after 40 min, then polished this block into a specimen of 3 mm (diameter) × 10 mm (length) by coring rig drill. As shown in Fig. 4.

2.3. CT scan procedures

Firstly, the sample was placed on a turntable to CT scan at the room temperature (20 °C); Then the atmosphere furnace bracket was moved to heat the sample in an atmosphere furnace; when it reached to the predetermined temperature, to get the full reaction inside the specimen, we need to continuously incubate it for 20 min, and then to remove the atmosphere furnace to CT scan the sample at the temperature. Then we can reheat and scan the sample of the next temperature. The heating temperature was set at 100 °C, 150 °C, 200 °C, 250 °C, 300 °C, 350 °C, 400 °C and 500 °C. The conditions of the CT scan test were a current of 70 A, a voltage of 70 V, magnification 105, the number of projection pieces 400, and superimposed frame rate 2fps. The scanning unit of resolution was 0.194 mm/magnification; experimental observation magnification was 105 times, and the size of the scanning unit was 1.8476 μm with distinguished aperture voids of 1.8476 μm. At each temperature, the scan time of the specimen was 30 min, and a total of 1500 slices were obtained. Then CT scanned images were reconstructed with the help of CT experiment analysis software. The evolution of the internal structure of anchoring material during the pyrolysis process was studied by analyzing its gray decay, 3D pore structure and the evolutional characteristics of the percentage of material corresponding to different attenuation coefficients.

3. Results and analysis of CT experiment

3.1. Gray scale attenuation analysis

Fig. 5 shows that the UPR anchoring material consists of powder particles, UPR cemented body and porosity at room temperature (20 °C). Among them, the powder particles were white with maximum gray scale, UPR cemented body was gray with lower gray scale and internal pores were black with zero gray scale. These three media is distributed alternatively with anisotropic properties.

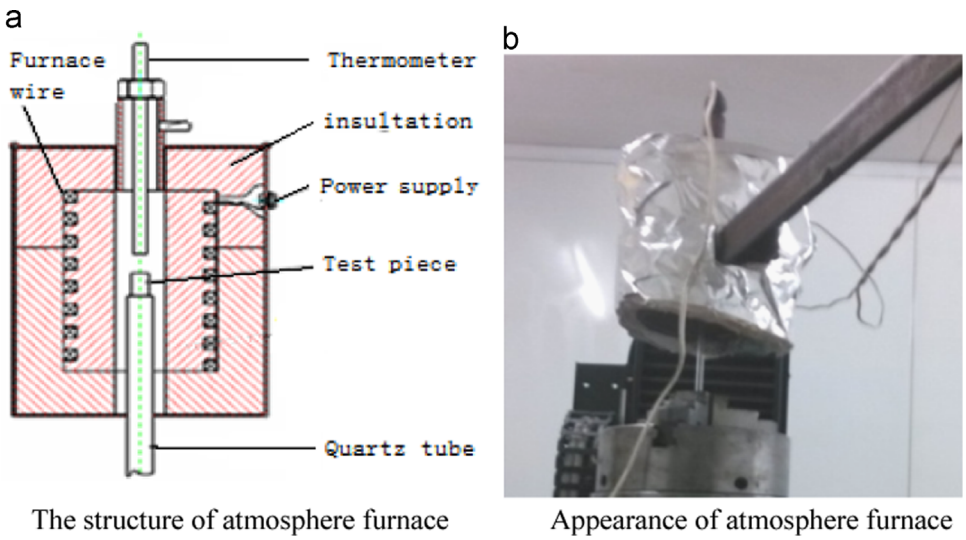

Fig. 2. Heating equipment of CT test. (a) The structure of atmosphere furnace (b) Appearance of atmosphere furnace.

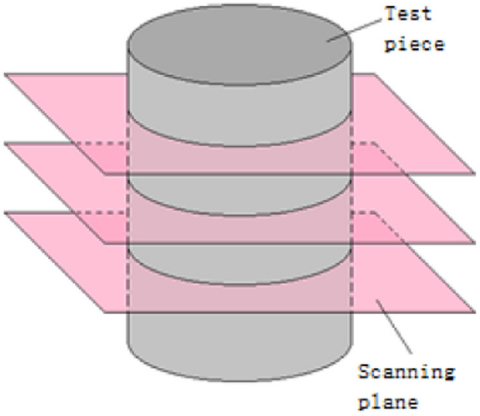

Fig. 3. Schematic of CT slice.

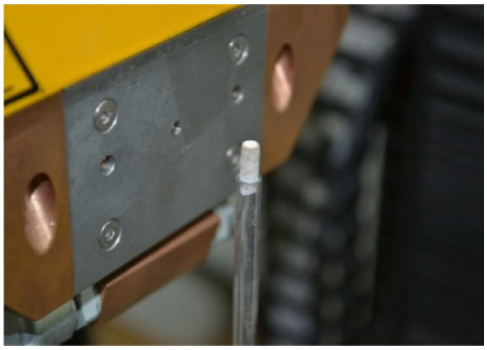

Fig. 4. Test block for CT scan.

To use the 500 layer plane maximum inscribed circle as a statistical area, we obtained the statistical results of gray scale in the area at different temperatures and plot the picture of average gray curves versus temperature, as shown in Fig. 6: the average gray was 5.68 at 20 °C, when the temperature rose to 100 °C, the gray was reduced to 5.66 due to evaporation of water inside the curing agent (the original curing agent containing a certain proportion of mixing water); then it increased with increasing temperature and reached its peak of 6.04 at 250 °C, increased by 6.3% compared to the normal temperature. The analysis considered that as the result of the completely cured of the resin. The average gray value of 250–350 °C was decreased, which indicated that the resin began to decompose. The figure of gray scale decreased significantly after 350 °C, and it dropped to 5.09 at 400 °C with a decreasing of 15.7% of attenuation compared to the peak. It dropped to 4.79 at 500 °C with a decreasing of 20.7% of attenuation compared to the peak. These results indicate that UPR pyrolysis and carbonization intensify at 350–400 °C, and the density decreases significantly while the pores increase obviously. The pyrolysis and carbonization tends to be complete at 500 °C. The correlation analysis showed that the glass transition temperature of the UPR anchoring material is about 250 °C.

3.2. 3D structure analysis

As shown in Fig. 7, in order to have a more direct visual representation of the evolution of the UPR anchoring material internal pore space structure, we achieved the overall structure of the 3D reconstruction by composing the cross sectional views of the CT scan sequentially [22]. The 475th–525th slice was selected in each temperature for reconstruction, and the middle part of each layer was selected to construct the $399 \times 399 \times 50$ pixel cube area, as shown in Fig. 8.

According to the changes in pore structure as shown in Fig. 8, the internal pore material did not change significantly in the range of 20–350 °C, and the porosity showed a slight increase. But the number of black pore increased significantly after 350 °C as well as the volume of the main gap. It proves that the thermal decomposition of UPR intensified after 350 °C and the internal pore expansion accelerated.

3.3. Evolution of pore group

In order to have a more comprehensive analysis of the evolutionary characteristics and the parameter change of the internal pore group of the material, the largest central region of the CT image plane was selected for the 3D reconstruction to build a cube with the pixel of $399 \times 399 \times 300$. By two-dimensional processing for 3D digital model, pore pixels can be segmented from the test piece. Based on the statistics of pixel point, the characteristic parameters of the pore group can be obtained, and it includes the number of the pores group, the size of the pores group, the size and shape change of the largest pore group, etc.

Fig. 9 shows the variation of the number of pore group with temperature. Based on the figure, the number of pores group was

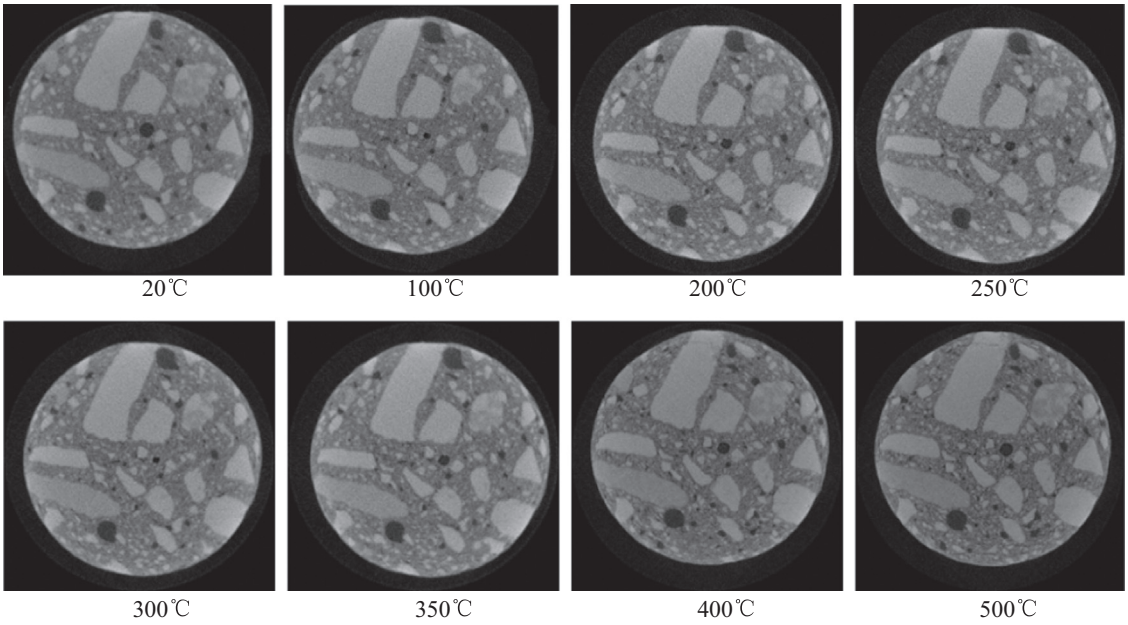

Fig. 5. The scanned images of Dir 500 plane of the layer at different temperatures.

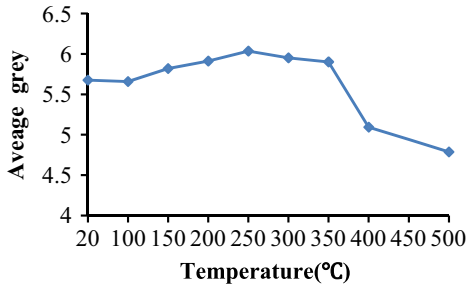

Fig. 6. Changes in average gray of Dir 500 layer plane at different temperatures.

4.7×10^4 at 20 °C, and it reduced to 4.1×10^4 at 100 °C, but it increased to 4.9×10^4 again at 150 °C, it is believed that this is the joint result of the evaporation of water in the pores and the thermal expansion stress that leaves small pores closed; in the range of 150–350 °C, the number of groups has no significant increase, but it increased to 6.9×10^4 in the range of 350–500 °C with an increase rate of 47.5%.

Fig. 10 shows the change of the size (pixels) of the pore group within the material with temperature. Based on the figure, the pore size of the group increased steadily from 6.9×10^5 to 10.3×10^5 in the range of 20–350 °C, but the pore size of the group increased to 20.8×10^5 in the range of 350–500 °C with an increase rate of 201.5%, and it shows that the thermal solution is obvious after 350 °C, the porosity increased significantly.

In order to show the change of the internal pore structure of the material visually, we used a small cube unit with the side length of 1 pixel to replace the pore corresponding to the pixels. To interconnect a number of porosity pixels to form a pore group, we can draw the distribution shape of the maximum pore group in 3D space by counting the pixels number of the area. Fig. 11 shows the shape changes of maximum pore group inside of 3D construction region within the material. The figure shows that the maximum pore group did not have an obvious shape change when it was lower than 350 °C, but the maximum pore group shape was significantly larger at 400 °C. Analysts believes that this is due to the pyrolysis of the resin which led to the rapid expanding of the pore group and the connection with the surrounding pore group between 350 °C and 400 °C. Statistics show that the pixel size of the maximum pore group increased from 4.5×10^5 to 6.8×10^5 between 20 °C and 500 °C, in another word, the maximum pore group's actual volume

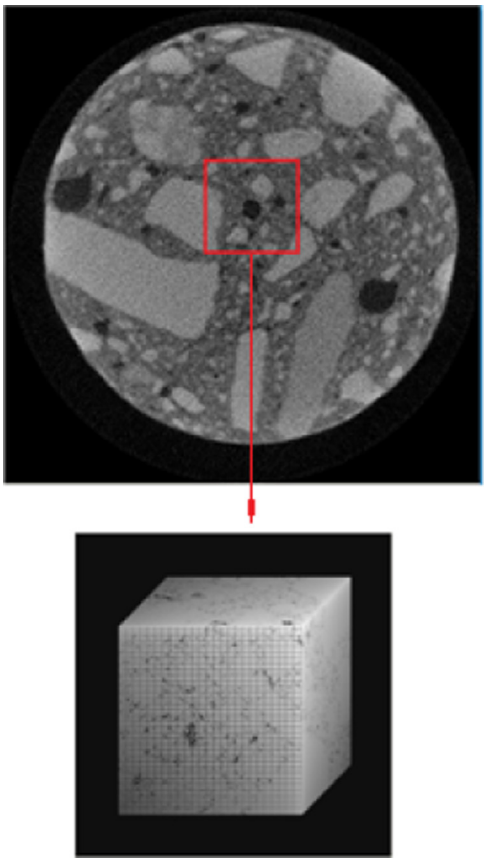

Fig. 7. Schematic of 3D reconstruction region.

increased from 2.8×10^6 μm^3 to 4.3×10^6 μm^3 with an increase rate of 53.6%.

3.4. Pyrolysis characteristics analysis of UPR anchoring material

Different attenuation coefficients are corresponding to different densities of matter. To do the statistical distribution of the number of the points corresponding to different attenuation coefficient in the constructed 3D cube with the pixel of $399 \times 399 \times 300$, and to divide the attenuation coefficient corresponding to different substances within the cube into 11 sections from small to large, Fig. 12 can be obtained based on the percentage of the different section corresponding to different attenuation coefficient. Different curves correspond to different temperatures in the figure. Fig. 12 shows the percentage of each attenuation coefficient corresponding to the pixel of a normal distribution internal anchoring material, indicating a homogeneous distribution of the content of each substance.

Based on the ingredients of UPR anchoring material and the correspondence of the attenuation coefficient with the density of matter, together with the help of segmentation method, the attenuation coefficient in Fig. 11 was divided into three sections corresponding to different media. The range of 0–0.0163 corresponds to porous medium, the range of 0.0163–0.0373 corresponds to the organic cemented body, and the range of ≥ 0.0373 corresponds to the hard and compact inorganic. Meanwhile, the curve of the three segments corresponding to the pixel cumulative percentage versus temperature was obtained, as shown in Fig. 13.

Fig. 13 shows that, the content percentage of the pixel dots of the attenuation coefficient in each section changed as the temperature increased in the range of 20–100 °C. In the range of 0–0.0163 zone (pores), the content percentage of the pixels decreased slightly. Analysts believes that it was mainly due to the expansion of the solid material caused by thermal stress which leads to the pores' compression; In the range of 0.0163–0.0303 zone (organic cemented body), pixels percentage reduced significantly, and analysts believes that it was mainly due to the evaporation of the water in the UPR anchoring material curing agent. The percentage of pixels in the ≥ 0.0373 zone (hard and compact inorganic) has a corresponding increase significantly.

In the range of 100–150 °C, the content percentage of the pixels increased slightly in the 0.0163–0.0303 zone (organic cemented body). Analysis shows that the UPR anchoring material is an organic polymer composite material. Its main components are the unsaturated polyester resin, which are a diol and an unsaturated dibasic acid polycondensation at high temperature having an ester bond and an unsaturated double bond linear polymer. The main chain contains double bonds of polymerization; they copolymerize with various kinds of vinyl monomers in the influence of the initiator at room temperature or slightly heating (generally below 150 °C) conditions, as shown in Fig. 14. The curing agent contains a small amount of organic peroxide compound (R–O–O–R) and aliphaticazo compounds(R–N=N–R'), and some of them have the chemical bond between 105 and 150 kJ/mol [23]. Therefore, when

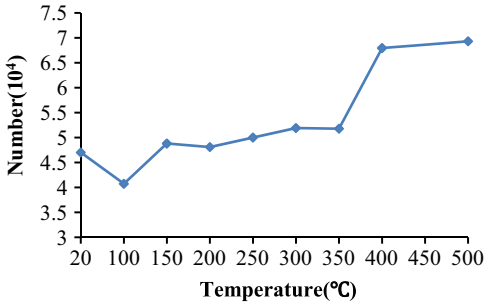

Fig. 9. The number of pores group variation with temperature.

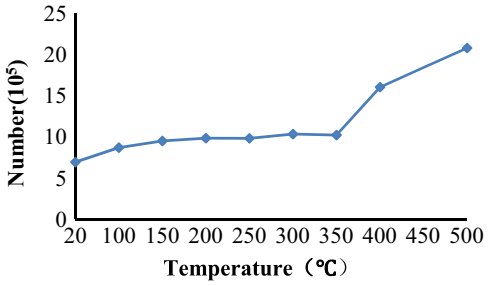

Fig. 10. Total pore group size variation with temperature.

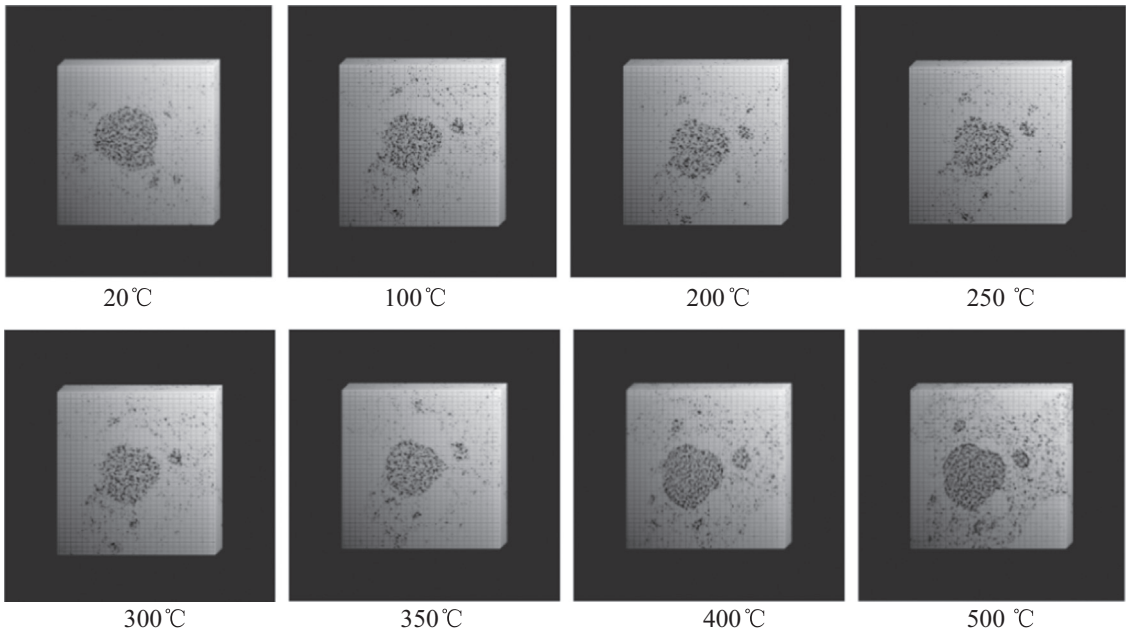

Fig. 8. Changes of $399 \times 399 \times 50$ cubic at different temperatures.

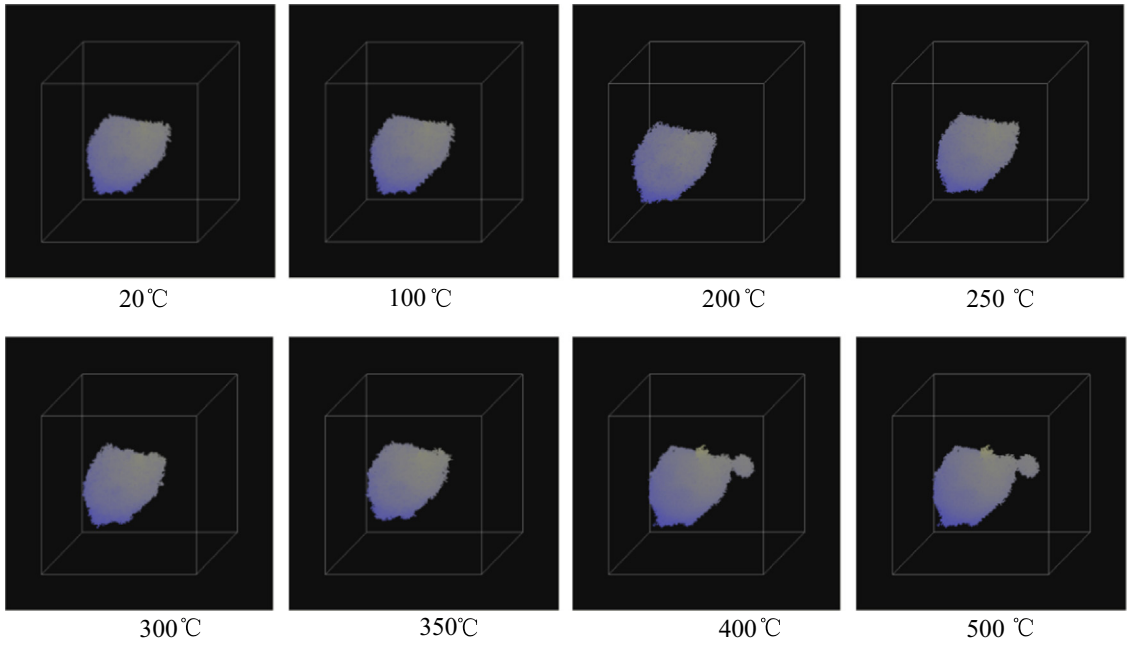

Fig. 11. Developments and changes of maximum porosity in the temperature in each group inside of 399 × 399 × 300 cube.

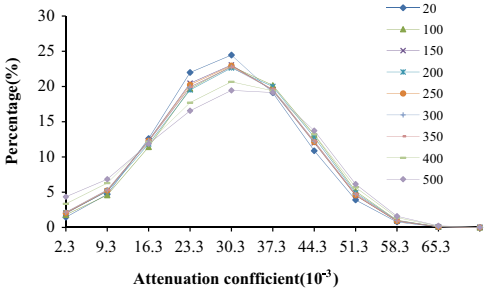

Fig. 12. Distribution of the different attenuation coefficients at different temperatures.

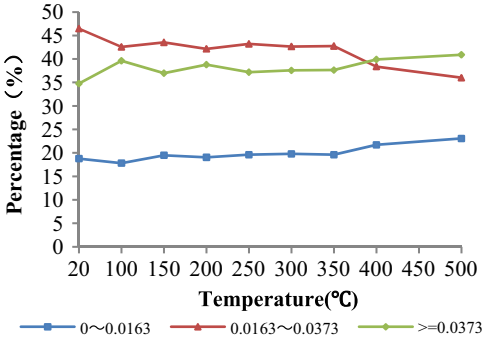

Fig. 13. The cumulative percentage change of the three different attenuation coefficients stages at different temperatures.

the temperature raises to 50–150 °C, these chemical bond will decompose to form a more stable chemical bond so that the anchoring material will be more fully cured, and the bond of the powder particles become closer.

In the range of 150–350 °C, the changing of the each section of internal anchoring material was relatively stable and the porosity increased slightly, which is the result of the curing effect of the resin that tends to be complete and a small amount of unsaturated resin decomposition.

In the range of 350–500 °C, the internal anchoring material pixel point content of each section experienced a sudden change,

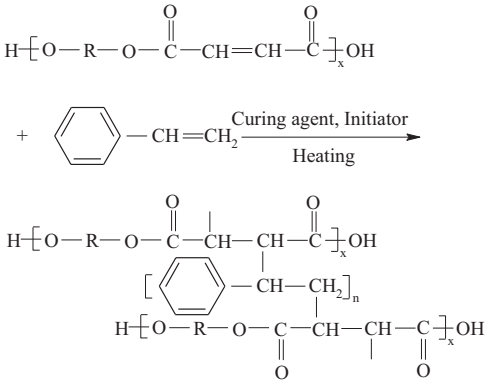

Fig. 14. The formula of crosslinking polymerization reaction.

and the percentage increased significantly in the range of 0–0.0163 and ≥0.0373 while that of the range 0.0163–0.0373 decreased drastically. This is due to the huge number of organic hydrocarbons in the UPR anchoring composite materials, which contains a large number of C–C bond with the bond energy of about 380 kJ/mol. Due to the presence of inorganic fillers, to promote its decomposition to generate free radicals by heating requires a high temperature of above 350 °C. Therefore, a large number of unsaturated polyester resin thermal decompose and oxidase after 350 °C which generates gas and the inorganic products and produces a large amount of porosity. When the temperature increases to 500 °C, the unsaturated polyester resin continues to break down, but the magnitude of changes decreases gradually to none until the unsaturated polyester resin was completely pyrolysed. Compared to the pyrolysis characteristics of pure UP that pyrolyses at about 300 °C [24], the thermal properties of anchoring composite materials is been enhanced when filled with inorganic.

4. High temperature mechanical property of UPR anchoring material

The mechanical stability of the anchoring material plays a decisive role in the application [25–28]. The mechanical properties

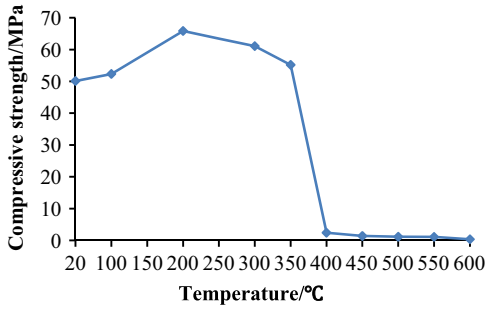

Fig. 15. Compressive strength change with the temperature.

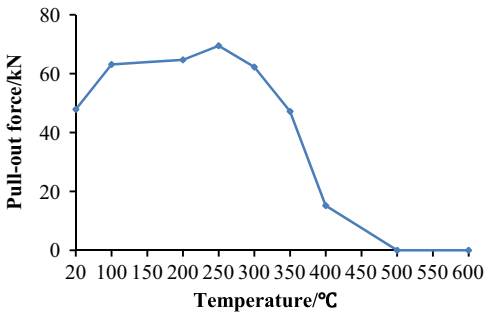

Fig. 16. Pull-out force change with the temperature.

of the UPR anchoring material changes with the microstructure under high temperature. Therefore, the high temperature mechanical properties of unsaturated polyester resin was studied by high temperature compression test and high temperature pull out test.

4.1. High temperature pressure experiment

The specimen for high temperature experiments were blocks of 70.7 mm × 70.7 mm × 70.7 mm. There were 10 groups of blocks with 3 specimens in each group, and molding process take MT146.1-2002(CN) as the reference. Firstly, put test blocks of each group into the XRMF-9X-efficiency energy-saving muffle furnace for heating. The temperature were set to 20 °C, 100 °C, 200 °C, 300 °C, 350 °C, 400 °C, 450 °C, 500 °C, 550 °C and 600 °C respectively, and the heating rate of 10 °C/min. Due to the large size of the specimen, the heat preservation for 40 min was required for the full reaction. After the specimens were natural cooled to the room temperature, the uniaxial compression experiments were carried out with the WAW-600 computer control electro hydraulic servo universal testing machine with a loading speed of 0.008 mm/s. After that, the compressive strength of specimens under different temperatures after high temperature was obtained.

Fig. 15 is the change curve of the compressive strength of the anchoring agent test block at different temperatures. According to the figure, the compressive strength was 50 MPa at room temperature, and the intensity peaked at 66 MPa when it was 200 °C with an increase rate of 32%; After 200 °C, the intensity began to decrease, and it dropped significantly after 350 °C. The compressive strength was only 2.4 MPa at 400 °C, which decreased by 95% compared to the room temperature; the compressive strength was 0.36 MPa at 600 °C, which decreased by 99% compared to room temperature.

4.2. High temperature pull-out test

There were 8 groups of blocks with 3 specimens in each group, the reference of the molding process ia MT146.1-2002(CN). The pull-out test specimens is composed of three parts, which are the pipe of the simulation drilling, the UPR anchoring material and the bolt. The bolt is the thread steel with the diameter of 16 mm and the length of 90 mm. The inner diameter of the simulation drilling pipe is 32 mm, and the length is 50 mm. The filling length of UPR anchoring material is 50 mm. Firstly, put test specimens of each group into the XRMF-9X-efficiency energy-saving muffle furnace for heating, and the temperature were set to 20 °C, 100 °C, 200 °C, 300 °C, 350 °C, 400 °C, 500 °C, and 600 °C separately, and the heating rate is 10 °C/min. After 20 min of thermal insulation, take out the test piece after an adequate response, then natural cooled them to room temperature for anchoring material using MLJ-70 type anchor pull test gauge.

As shown in Fig. 16, pull-out force of resin anchoring material at room temperature was 47.9 KN, and the pull-out force increased as the temperature increased, reaching to the maximum point at 69.5 KN at 250 °C with an increase rate of 45.1% compared with room temperature; then the pull-out force decreased with the increase of temperature, and it dropped to 47.2 KN at 350 °C, which was decreased by 1.5% at room temperature. However, mutations occurred at 400 °C, and the anchoring material burst and partly detached in the high temperature with the pull-out force reduced to 15.2 KN, which decreased by 68.3% compared with room temperature. When it was at 500 °C and 600 °C, the material was completely carbonized and detached, and the pull-out force was lost.

5. Conclusion

With the help of CT image reconstruction, we have analyzed the decay of gray, 3D pore structure during the pyrolysis and the mechanical properties of UPR anchoring material at different temperatures, and the results are as follows:

(1) Resin anchor material has the features of significant thermal decomposition and instability at high temperatures. In the range of 20–300 °C, the average gray fluctuates slightly and it peaks at 6.04 at 250 °C. In the range of 350–500 °C, the average gray has an attenuation of 20.7%. It shows that there is a huge amount of pyrolysis in the internal organic of the anchoring material resin after 350 °C, and the density of the material decreases obviously.

(2) The inner pore increases significantly and each of the pores connected gradually to form a larger pore group as the pyrolysis of resin anchoring material increases at high temperature. In the range of 20–300 °C, the pore group in the construction region increases slightly, but the number of pore group experiences an increase of 47.5% in the range of 350–500 °C, and the size of pore group increases 201.5%.

(3) The percentage of various substances in anchoring material corresponding to different attenuation coefficient shows a normal distribution at room temperature. The change pyrolysis of different material in the anchoring material at high temperature is obviously different. The UPR cemented body reduces significantly as the temperature increases. The pore enlarges as the temperature increases and the hard and compact inorganic increases significantly as the temperature increases.

(4) UPR anchor material's mechanical properties are significantly attenuated under high temperature. The compressive strength peaked at 200 °C with an increase rate of 32% and it dropped significantly after 350 °C. The compressive strength at 400 °C decreased by 95% compared to room temperature. Pull-out strength of resin anchoring material reached to the maximum

at 250 °C with an increase of 45.1% compared to the room temperature. Pull-out strength decayed faster after 350 °C with a rate of 68.3% compared to room temperature at 400 °C, and the pull-out strength is lost at 500 °C and 600 °C.

The microstructure variation of UPR anchoring material at high temperature obtained by CT scanning experiment agrees with the experimental results of high temperature mechanical properties.

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