

Reparation and properties of ceramifiable ethylene propylene diene monomer

Rubber composites reinforced with chopped polyimide fibers

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Abstract: The ceramifiable ethylene propylene diene monomer (EPDM) composites with different fiber contents were prepared by taking EPDM as the matrix , Kaolin and talc as the functional fillers , Aluminum Hydroxide as the

Flame Retardant , The and the chopped polyimide (PI) fiber as the reinforcement. The effects of chopped polyimide fiberson the tensile properties,Thermal Stability and microstructure of ceramifiable PI Fiberkaolin-talc-akoh)3/ EPDM (PKTA/EPDM) composites were investigated, and theceramicization Mechanism of chopped polyimide fiber reinforced composites was analyzed. The results show, the tensile strength of the composites decreases with the increasing of chopped Imide fibers. When the chopped PI fibers are less than : [mass ratio to

EPDM),Mechanical properties of the composites are. The ceramifiable PKTA/EPDM composites can be ceramicized after pyrolysis at 800-1 100C. The addition of 4 : ,8 : mass ratio to EPDM chopped poly. imide fibers can effectively keep the shape stability of the Composites after pyrolysis at the.1 0 C , and thebending strength of the ceramic productf I between 6-18 MPa. The results of thermal analysis show that the thermal

LIU L d,qin y,song J q,et Al Preparation and properties of ceramifiable ethylene propylene diene monomer rubber Composites reinforced with the chopped polyimide iibers[j]. Acta materiae Compositae sinica,2017, : 2800-2809 (in Chinese).

and SEM Analysis I ndicate that the pyrolysis and carbonization of the chopped PI fibers in the char layer form the Structure of fiber^reinforced char layer. The PI fiber reinforced structure contributes to obtaining the well-shaped EPDM Composites.

Keywords: Ethylene Propylene diene monomer (EPDM) ; chopped polyimide fiber ; Composites thermal stability ;

Ceramic mechanism

can be Tao Ceramic polymer matrix composites are _ New blocking burn , Thermal protective material , has been widely used in the field of passive thermal protectionGeneric Research and commercialization application □ . vs. traditional thermal protection aggregate object matrix composites , These materials are not only good at room temperature Good mechanical properties , and in high temperature aerobic environment , to convert to self -supporting Tao porcelain , also has a certain intensity and can maintain the size of the basic stability of the .

Currently, the research of Tao Ceramic polymer matrix composites is mainly with silicone polymer as matrix , add

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clay minerals to porcelain fill , Low melting point structural control agent or flux-prepared silicon base tao porcelain

composites. The main chain of silicone polymers is one Si-O-Si key, side chain is alkyl, Vinyl, phenyl Organic base. Silicone keys have high key energy, Thermal stability significant. and and thermal cracking mechanism and pyrolysis kinetics of silicone Polymers The study of is also getting better. , for silicone polymer composites Tao Research provides theoretical support [813]. Cheng Y B etc [A]] first proposed can be porcelain The concept of the and the porcelain cloud parent / Silicone Rubber Composites conducted a series of studies , proposed silicon rubber composite tao porcelain mechanism . high temperature silicone polymer crack after solution product is network-like structure Si O₂ , can be used with mineral porcelain fillers occurrences of molten eutectic reaction ,The molten fluid that the meeting gradually seeps into , the effect of bridging between filler particles [1-15] , ,, but this eutectic anti higher temperature should occur , Tao Porcelain products with low strength . to add mica For example , Mica particles and silicone rubber cracking products SiO₂ in the 1 ,, °c occur when the eutectic reaction produces liquid phase the [14-15]. for this , on To add a melting-point flux or structure control to the system Agent , to reduce Tao ceramic reaction temperature , Maintain dimensional stability and mention Gao porcelain product strength [on] .

differs from silicone polymers , about carbon-based polymers can be porcelain The Study is relatively small .. due to the carbon base polymer main chain is mostly made from carbon chain composition , release CO₂ [] under high temperature aerobic conditions or C O Gas body , leaving amorphous carbon , its pyrolysis residue rate affects the most final porcelain performance . has literature report , phenolic tree with high residual carbon rate Grease as a base-prepared ceramic resin matrix Composite in the , Cstarts to generate Tao porcelain layer , Add functional lightweight on this basis hollow beads , Reducing material density , Improve material insulation to [to] . and a carbon-based polymer with a low residual carbon rate as the matrix , porcelain performance affected by residual carbon rate and amorphous carbon layer , Final effect lower than the silica substrate performance [21-22] , The restricts the carbon-based system Tao apply to porcelain . also , have research findings , in glass / Poly b The addition of chopped glass fibers in the alkene system is easier to form a continuous cause secret body [A] . Di wait [] with ethylene Jg acid vinyl ester copolymer (EVA) is the matrix , on add glass , Organic Montmorillonite , triple On the basis of cyanuric acid salts, also added chopped glass fiber , system to have a very good flame-retardant properties of the porcelain EVA Composites .

for EPDM rubber (EPDM) for ceramic of the matrix three meta EPDM composites during pyrolysis , matrix pyrolysis residue low , after pyrolysis carbon layer instability causes Tao porcelain products to appear fragments ,, and Tao Porcelain products with low strength , in EPDM is a matrix , High Ridge tu (kaolin) and talcum powder (talc) to porcelain filler , Al (OH)₃ on the basis of a flame retardant , add different levels of chopped polyimide fiber (pifiber) The is modified as an enhanced material . PI fiber has good mechanical properties at elevated temperatures , and fire does not burn burn , after softening to form fibrous charcoal layer . PI fiber with EPDM The composite can improve the strength of the carbon layer during the pyrolysis of the material. , final to obtain a good ceramic properties of the porcelain PI Fiber -kaolin ^ talc - Al (OH)₃ / EPDM (Pkta/EPDM) Composite . use muffle to get the material under air ~ 1 C Tao Porcelain Products , using thermal analysis , SEM To research Tracing Analysis of the generation mechanism of Tao porcelain residues , Analysis of composites in high temperature tao reaction process , Research short cut PI Fiber to porcelain pkta/ Effect of EPDM composite on high temperature performance .

1. Experimental materials and methods

1.1 raw materials

EPDM (EPDM), Grade 4045 M , Third list body-B-down borneol (ENB) , content is 7 6 WT % , Ethylene content to wt % , Mitsui ; Zn O , AR , Chemical Reagents ; Hard fat acid,AR , Chemical Reagents ; diphenyl sulfide (DM) , AR , Chemical Reagents ; Liquid Paraffin , AR , Chemical Reagents ; gas-phase white carbon black , specific surface area m² / G , Shanghai Aladdin Reagent Limited Division ; Sulphur , AR , Chemical Reagents ; Polyimide chopped fiber (without over surface processing) , length 3 mm , new material for Jiangsu Aoyuan Division ; High temperature calcined kaolin , size One pm , Shanghai Aladdin try Agent Co., Ltd. , chemical composition See table 1; talc , size Ten

~ pm, Hubei Xing Yinhe Chemical Co., Ltd., chemical composition See table 2; AK 0 H), AR, Chemical Reagents; Glass Powder, particle size Ten ~

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pm, Soften Point ~ 830 °c, Guizhou Witton Crystal phosphate electronic material Material Co., Ltd., chemical composition See table 3.

1.2. Sample Preparation

Apply a vacuum drying box to the kaolin, talc, Glass in 12C conditions Dry treatment h. by formula EPDM and fill material add two-roll mixer for mixing, to be mixed after, To remove the film, Place H above. then counter slice, load, sulfur to. The pressure during vulcanization is ten MPa, vulcanization temperature 160C, vulcanization time, min. by Table 4 Formula preparation can be porcelain pkta/epdm composite material.

1.3 sample pyrolysis

after vulcanization, the composites are cropped to mmXMmX 3 mm (, Add crucible to alumina Tao porcelain interlayer, sample down-shop _ layer Alumina powder, then put the crucible in the Muffle in. temperature control programs to, C, 900C, 1 000 C, 1100c, in air condition, samples in 4 temperature segment Insulation min, Then remove the sample and cool it to room temperature for performance test.

1.4 Performance token

kaolin, talc, The composition of the glass is made up of Holland pana - lytical B. V. Company's axiosadvanced X x-ray fluorescent light

Spectrometer for analysis tests. tensile of composite at ambient temperature can use CTM 6001 microcomputer controlled electronic universal material Tester Enter Line test, based on GB/ T528-2009 Standard □, Extrude rate to \$ mm/min. Composite thermal stability test using Germany country Netzsch Company's high temperature TG-DSC Comprehensive Thermal Analyzer, in an argon atmosphere, Heating rate is 0 k/min, temperature range to ambient temperature ~1 C. to Composite in 4 temperature Section high temperature pyrolysis of cooled specimens with flexural strength and cross-section Micro profile. where Tao Porcelain product section microscopic appearance in Japan Electronics Co., Ltd. model is JSM - IT scanning electron microscopy to divide analyze. bending strength INSTRON-1341 on Three-point Bend method "" test, load rate is 2mm/min. tao porcelain "" macro-shape photography with digital camera.

2. Results and discussions

2.1 can be porcelain Pkta / EPDM extrude Performance

can be porcelain PKTA/EPDM composites tensile at ambient temperature can be as follows 1, . know, composite with different fiber content Pull strength on 3. 5 ~ 7. 0 MPa between, break elongation at 100% ~800%. with PI Increase fiber content, anti-pull strength and elongation at break when no PI Fiber is added,

The tensile strength of the composite reaches 6.5 MPa above, Break elongation rate up to 700% above. when chopped PI fibre content increasing when, chopped fibers increasingly difficult to disperse evenly in the rubber matrix, Many chopped fibers may squeeze together, Fiber spacing distribution non -Uniform, even has a bend, , back-fold

status, These conditions all cause vulcanized rubber internal interface defects increased, Which in turn causes A duplicate the material to break earlier in the stretching process [1]-2. when PI Fiber /EPDM quality ratio 10:100 when, at this time composite pull extend strength to 4 MPa below, break elongation is more drastic down down 200%below.

2.2 porcelain pkta/ EPDM macro appearance after pyrolysis

can be porcelain of different fiber content Pkta / EPDM Composite Material material on ~ 1 100°C macro topography after pyrolysis figure 2 is shown in . unknown, on i~ 1 100° C range pyrolysis, Composite all issued gave birth to varying degrees of Tao porcelain reaction, generated Tao tiles. with temperature degrees high, Significant volume contraction occurred after pyrolysis of composite materials. when not adding a short cut PI Fiber, composite pyrolysis all occurrences Varying degrees of Curl, broken into fragments at the same time. when PI Fiber / EPDM quality ratio 2:100 time, after pyrolysis of composites still a slight curl and a crack on the surface, but not broken split into dispersed block, to keep specimen complete shape. when PI Fiber/EPDM quality to 4 : ~ 8 :, Composite The curl and surface cracks in the material after pyrolysis are basically gone. , Same to keep the specimen shape intact. when PI Fiber /EPDM quality The measure is 8 :, Composite surface is more flat after pyrolysis, dimensional stability better. But when PI Fiber/EPDM quality ratio to 10:100 when, sample on i C and 1 \$ C pyrolysis samples appearance significant difference. The May be on the one hand because the composite material has a layer up and down during pyrolysis. Ai 0 3 Uneven powder causes heat Solution temperature uneven, On the other hand may be chopped fiber content than more, Sample pyrolysis prevents the diffusion of molten liquid phase from causing the tao the degree of porcelain is significantly wider. with 1C Pyrolysis samples For example, different sample locations cause different pyrolysis temperatures, again cause pyrolysis Color difference after (Chart 2 (F)). in center color black area field, lowest degree of pottery, can also observe matrix material cracking Black carbonized layer after solution (as A point), in the surrounding edge position (as Cpoint T), Tao the highest degree of porcelain. between the two is a white color transition area (such as bpoint T), Tao is also two degrees of porcelain between people.

2.3 can be porcelain PKTA/EPDM flexural Performance after pyrolysis

can be porcelain of different fiber content Pkta / EPDM Composite Material material on ~ 1 [C after pyrolysis tao Porcelain products flexural strength as table 5 is shown in . unknown, when PI Fiber /EPDM quality to (0~ 2) : when, cannot maintain shape integrity after pyrolysis of composites and Dimension stable, No bending strength test. when PI Fiber/EPDM quality ratio Ten : when, composites C-5 due to partial sample pyrolysis temperature heterogeneity and experimental error and other factors causes greater bending strength. when PI Fiber /EPDM quality is (4 ~ 8) : when, product bending after pyrolysis of composite materials intensity in 6~ MPa. also, pyrolysis temperature samewhen, composite pyrolysis product flexural strength increases with fiber content To increase (C -5 except). When the amount of fiber added is the same, , compoundmaterial in i C and 900 C The flexural strength after pyrolysis is quite, Change less, on 1 C The flexural strength increases quickly after pyrolysis (C -5 except). as composite material C-2 in 1 \$ C Thermal bending strong degrees compared to 900C pyrolysis added 9, composite C -3 in 1 C The flexural strength after pyrolysis is compared to 900 CAdd after pyrolysis plus ? 7, Composite C -4 The flexural strength after pyrolysis is compared to 900C after pyrolysis increased ? 0%. and composite materials in

1 C pyrolysis product flexural strength and 1 C after pyrolysis product The flexural strength is substantially equivalent to. presumed to be below 1 C when porcelain padding participation low level of ceramic reaction, The pyrolysis product has a _ The fixed strength of the is due to the diffusion of the glass material into the pyrolysis carbon layer after melting. Liquid Bridge Structure. and above 1 Strong bending after pyrolysis degrees increased rapidly may be due to the ceramic filler involved in the Tao porcelain, reaction.

2.4 can be porcelain PKTA/EPDM Thermal stability

diagram 3 is EPDM and PI Fiber under Argon atmosphere conditions thermogravimetric analysis (TG) and differential hot weight (DTG) curve. can look at go to, The thermal stability of the PI fiber is significantly greater than

that of EPDM . EPDM in argon atmosphere conditions only _ weightlessness phase , its thermal decomposition temperature degree range is very narrow , on 464.5 °C Zero weightlessness rate at , on 495.3 °C Slow quality loss , at this time the rubber carbonization basic end . and PI Fibre in argon atmosphere conditions there are two levels of weightlessness

Segment , First stage weightlessness 34%, fiber from 577° C starts the decomposition , on 601.7°C maximum decomposition rate is reached , in 687° C weightlessness The rate significantly drops . hereafter , temperature rise , Fiber second phase loss heavy rate significantly less than the first stage , until 1 v C when fiber pyrolysis residue rate remains greater than . 7%. diagram 4 for chinaware pkta/EPDM TG for composite materials under Argon atmosphere conditions

curves and DTG Curve . from the diagram 4 The thermal parameters of the composite material "" number as table 6 is shown in .

from Diagram 4 (a) to see , can be porcelain of different fiber content Pkta / EPDM The cracking process of composite material can be divided into three phase : first stage from initial temperature ~ C , Weightless about 3%, This stage composite system Al (OH) and vulcanization aids

agents such as heat release of small molecular substances appear thermal weightlessness ; Second-order The segment occurs on the ~ 700 °c, composites cracking most plays Strong , weightlessness approximately 45%. This phase EPDM Matrix and PI fibrous hair live cracking release mass CO ,H₂O,C O₂ etc gaseous small molecules quality (third stage) ~1 v C , This phase of composite heat solution residues decrease and tend to stabilize, on 1 \$ C when guaranteed hold greater than 49%. from Diagram 4 (b) can see ,add fiber before and after Maximum decomposition temperature and thermal decomposition rate of composites no clear significant difference . Union diagram 4 and table6, after fiber composites Initial decomposition temperature and pyrolysis residue rate slightly increased , indicates that PI The addition of the fiber can improve the thermal stability of composite materials .

Z 5 can be porcelain PKTA/EPDM The microscopic topography of the cross-section after pyrolysis porcelain pkta/epdm composite C - 0 , C - 4 , C -5 The microscopic topography of the cross-section after pyrolysis is as follows 5, diagram 6 and Diagram 7 shows . know , without chopped fiber , composite material C-0 A continuous liquid phase structure occurs after the pyrolysis of 900 C , But the section still has a large number of not yet To participate in the reaction of the ceramic filler particles exist (Chart 5 (a)). glass material softening point range to 550 ~830C, speculate at this time of continuous the liquid structure is the glass melts after the diffusion of the formation of the . and in

temperature/ C

diagram 4 can be porcelain PKTA/EPDM Composite in argon atmosphere TG (a) and DTG Curve (b)

Fig. 4 TG (a) and DTG (b) Curves of ceramizable PKTA/EPDM composites in Ar atmosphere

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1 000 ° C after pyrolysis the continuous liquid phase structure becomes more compact and even , porcelain filler particles are basically gone . (Chart 5 (b)), at this time , The formed continuous liquid phase structure includes not only the liquid produced by the molten glass phase , and kaolin , talc and other occurrences of the Tao ceramic reaction formed by the Liquid structure . when PI FIBER/EPDM quality is 8:100 when , can be porcelain pkta/epdm composite material C - 4 in 900 C after pyrolysis, there is not only a large number of carbonized PI Fiber interspersed in Liquid structure , and a small number of holes exist (Chart 6 (a)). and double fitting material C -4 on 1 C pyrolysis with900 C the section after pyrolysis appearance completely different , This time the glass melts into droplets , Even possibly Initial vaporization of evaporation causes volume contraction and a small amount of porosity . But at this time the The the porcelain filler participated in the ceramic reaction , To make the overall ceramic structure moreHigh (Chart 6 (b)). This is related to composites with pyrolysis temperature increase will be issued Birth volume shrink and in 1 C The

flexural strength after pyrolysis is significantly Gaussian one to .

can be porcelain PKTA/EPDM Composite C - 5 in 1 C after pyrolysis, three distinct regions appear on the sample surface . these three

The microscopic topography of the area section is shown in the diagram 7 (a)~ 7 (c) shown . know , in Sample center position area (Chart 2 (f)) A point), Break surface does not form a continuous liquid phase structure , and mass dispersed liquid and oak carbon layers bonded together , Short cut fiber runs through the carbon layer to form a whole (**Figure 7 (a)**). in the transition area (2 (Fbpoint)) , pyrolysis temperature is greater than the center position , cross section liquid obvious increase , liquid phase spreads to carbon layer bonding to form continuous phase junction construct , The chopped fiber continues to play an enhanced role in the carbon layer. (Chart 7 (b)). in the sample edge area (2 (f) C Point , hot Solution temperature greater than transition region , When liquid phase increases, diffusion forms whole Short cut fiber reinforced continuous liquid phase structure of body (Chart 7 (c). addition , * 7 (c) and diagram 6 (a) similar appearance , guesses the sample C Point area pyrolysis temperature 900Cabout . composite C-5 in 1 C after pyrolysis the specimen cross-section is shown in Fig . 7 (d) shows . This temperature condition , the The porcelain filler has undergone a ceramic reaction. , Glass material Melt fluidity Good , Sectional topography of composite materials and graphs 6 (b) similar .

The matrix material in the ceramic silicone rubber composite can be cracked

(c) C point in Fig.2 (f) at 1 c

diagram 7 can be porcelain PKTA/EPDM Composite C-5 after pyrolysis of different temperature sections SEM Image

Fig. 7 SEM Images of fracture cross-sections of ceramizable pkta/epdm[composites C-5 after pyrolysis at different temperatures

to a continuous network-like Si O 2 skeleton Structure ^[a]. this Si O 2 The skeleton structure can remain relatively stable during molten liquid phase flow , plus Si O 2 to participate in the formation of the Tao ceramic reaction Liquid Bridge Structure to keep shape complete ^[14-15, 1]. vs. ceramic Silicone rubber composite materials material different , porcelain ternary EPDM rubber composites , base material material pyrolysis residue low , in inert gas 1 000° C only after pyrolysis left 3. 7% (diagram 3 (a), and a discontinuous non-is obtained after splitting network-like loose carbon layer structure . This loose carbon skeleton not only low intensity , and cannot maintain shape intact and dimensional stability , in melt Melt-liquid flow process breaks , Warp Curl , causes the final pyrolysis product cannot keep shape complete (2 (a)). When adding a shortcut PI after fiber , before molten liquid phase appears (temperature below glass material softening point C),EPDM has been cracked to form a loose no stereotypes carbon layer , at this time PI fiber not cracking () 3), with fiber -dimension shapes interspersed in carbon layers , helps increase the carbonization layer strength . with temperature rise to + C above , low melting point glass starts to appear molten phase , dispersed liquid and carbon layers bonded together , make lax The carbon layer begins to become tight . and at this time PI fibers start cracking

Solution , The fibers inside the composite are carbonized by oxygen-free conditions , PI maintain fiber shape after carbonization , enhances as with . at this time , carbonized fibers move the carbon layer through to form the whole carbon skeleton structure (similar to network structure) , causes the carbon layer to be in a large number of liquid phases keep stable during flow . then temperature rises to 900C, glass [] Increasing liquid phase after melting of glass materials , Melt Liquid Flow diffusion to form fiber reinforced liquid phase structure in carbon layer . hereafter , temperature rise to 1C and above , porcelain filler participate in ceramic reaction , improves pyrolysis product strength .

3. Conclusion

(1) do not add chopped polyimide (PI) High-alumina for fiber Ridge Soil (kaolin)- bone powder

(talc)-Al(OH)₃/ ternary epdm glue (EPDM) failure to maintain shape integrity after pyrolysis of composites, Add add short cut PI The fiber can improve the thermal stability of the composite and maintain The stability of the composite in the pyrolysis shape . with fibre content , compound material tensile strength and elongation at break continue to drop . when pifi ber/epdm Quality ratio (4~8) : at the end of the,composite not only good mechanical performance , on i ~1 (8)C shape relative after pyrolysis

Good , and the bend strength reaches 6 ~ MPa .

(2) to add a porcelain of the same fiber content PI Fiber kaolin-talc - Al(OH)₃/EPDM Composite on 1 °c when the flexural strength of the pyrolysis is compared to 900° Cpyrolysis Enhancement 70% above . binding section SEM Analysis , Composite at less than 1,C when hot The solution is mainly molten liquid phase diffusion to the carbon layer forming fiber Dimension Enhanced liquid structure , Bend Strength Low . Composite in 1C When the temperature is and above the porcelain filler will participate in the Tao reaction , Can obviously improve the bending strength of Tao porcelain products .

(3) with EPDM The is a matrix of the ceramic polymer base compound material , add chopped PI fibers in the matrix cracking process to the carbon the layer to the fiber enhancement , carbonized fibers run through the carbon at elevated temperatures layer to form a network-like carbon skeleton structure , the carbon skeleton structure can be keep stable during molten liquid phase flow , After pyrolysis, you can get the ruler inch stable , Shape Complete Tao Porcelain products .

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