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Nanocomposites Preparation Method Based on Bubbles Explosion and Nanocomposites Capability Evaluation Method Base on Fractal Theory and TEM Image

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1. Introduction

Spontaneous agglomeration tends to occur between nanometer scale particles for their high surface free energy. The size of particle agglomerates granules is always beyond nanometer scale. The spontaneous agglomeration of nanometer scale particles and high viscosity of epoxy resin solution made it difficult to disperse nanomaterials into polymer as the configuration of nanometer scale particles. How to achieve nanomaterials' symmetrical decentralization in epoxy resin by the configuration of nanometer scale particles is one of the "choke points" of polymer nanocomposite preparation. To solve the problem, lots of researching works have been developed by experts and many inaugurated results be achieved. Presently, there are many methods applied in the polymer nanocomposite preparation, such as intercalation polymerization, solgel, in-situ polymerization, ultrasonic method, blending, chemical dispersant method and so on.

In this chapter, a nanocomposites preparation system based on diminutive bubbles explosion is designed. By applying this preparation system, we can disperse inorganic nanometer material into polymers as the configuration of nanometer scale particles. Compare to traditional dispersion methods, the proposed method tends to obtain ultrahigh stretching rate and increase the freedom degrees of nanoparticles dispersion. As the particles aggregates serve as nucleation agents, and the bubbles expand on the surface of the aggregates, the nanoparticles would disperse more effectively into polymer without dispersant.

At present, there are many researchs and reports about preparation and performance evaluation of nanocomposites. Methods usually used to evaluate uniformity of nanoparticles are X-ray diffraction, transmission and scanning electron microscope. Detecting thermodynamics performance of nanocomposites is an indirect method for uniformity evaluation. Although these methods aforementioned are explicit, they can't satisfy the routine and fast examination request for production and application departments. And the application and promotion of nanocomposites has been affected to some extent. So, the quantitative evaluation method for size distribution and space distribution of nano-particles is essential for preparation and research of new nanocomposites.

To rightly evaluate the size distribution and space distribution of nano-particles, the exact Mathematical model must be established and the picking-up method for corresponding parameter should be found. Nanocomposites belong to the category of ultra fine powder. Researchs show that the size distribution of ultra fine powder has fractal characterization. Many irregular discrete objects in nature distribute in space as fractal. So, fractal geometry provide a good mathematics frame for this kind of complicated system. It should be regard as a irregular dispersion process to disperse nanomaterials into polymer. The TEM images of nanocomposites can be directly obtained by transmission electron microscope. In this chapter, we will propose a evaluation method for size distribution and space distribution of dispersed phase in nanocomposites based on fractal theory and TEM image. Applying the method to describe distribution of filling particles in the polymer composites, the results show that it can exactly evaluate the size distribution and space distribution of nano-particles.

2. Nanocomposites preparation method based on bubbles explosion

To obtain suspended dispersion of nano-particles in liquid without any dispersant, a novel epoxy resin/SiO₂ nanocomposites preparation method based on diminutive bubbles explosion is presented. And, corresponding nanocomposites preparation system was designed. The preparation system applies compressed gas as transmission medium to carry nanomaterials into epoxy resin solution. The compressed gas with nanomaterials turns into diminutive bubbles distributing in epoxy resin/SiO₂. The great pressure difference between inner and outer-bubbles led to bubbles inflation and explosion. During the bubble inflation, bubble oscillation may generate. The stretching rate may reach 10⁶s⁻¹, which favors more homogeneous dispersion of nano-particles. During the bubbles explosion the released energy and the explosion shock waves disperse the nano-particles into epoxy resin solution. By using the preparation system, epoxy resin/SiO₂ nanocomposites were prepared. The SiO₂ dispersed into epoxy as the configuration of 15nm - 30nm particles.

2.1 Preparation method

In preparing polymer nanocomposites, laminar flow mixing of the nanomaterials is brought about by the shearing and stretching of polymer solution. In the preparation method based on diminutive bubble explosion, the key variant s about nanomaterials dispersion are the shearing rate and stretching rate.

2.1.1 Stretching dispersion effect

According to Tadmor and Gogos's dumbbell model, under the stretching rate ε , in terms of granule size(r) and the polymer viscosity(μ) the maximum dispersion force exerted is given by

$$F_{\text{Max}} = 6\pi\mu\varepsilon r^2 \quad (1)$$

To separate the agglomerated nanomaterials granules, F_{max} must be larger than the interaction force between nanomaterials granules F_{M} . The interaction forces between nano-particles are mainly due to surface interactions. The action distance range of attractive forces between nano-particles agglomerated granules is about 50 nm. The intermolecular interaction between two granules with radius r at spacing h may be approximated to

$$F_M = \frac{Hr}{24h^2} \quad (2)$$

where H is the Hamaker constant. Then, we got the minimum granule radius that can be separated under stretching rate ε , it can be expressed as

$$r_{\min} = \frac{H}{144\pi\mu\varepsilon h^2} \quad (3)$$

Eq. (3) demonstrates that stretching is an effective means of separating agglomerated granules. Nano-particles can be dispersed into polymer with enough high stretching rate ε .

2.1.2 Diminutive bubble explosion dispersion method

When the diminutive bubble inflation in the liquid polymer, the liquid polymer around the bubble is subject to stretching, the stretching rate is

$$\varepsilon = \frac{1}{r_0} \frac{dr}{dt} \quad (4)$$

where r_0 is the initial radius of the compressed bubble. According to Zaremba-DeWitt model, we got the diminutive bubble inflation model, shown as Fig. 1.

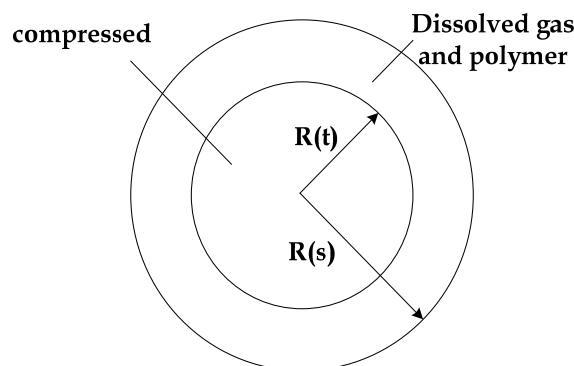


Fig. 1. Bubble and polymer shell

During the bubble inflation, the liquid polymer surrounding the bubble is subject to high stretching rate. When the bubble expands, viscoelastic deformation will occur in the surrounding liquid polymer. As the expansion process is very fast, a certain amount of the elastic potential energy is stored in the surrounding liquid polymer macromolecules because there is no time for energy release. Following bubble expansion, the gas pressure in the bubble decreases monotonically, whereas the elastic potential energy stored in surrounding polymer macromolecules increases. When the elastic storing force of the liquid polymer surrounding the bubble becomes larger than the bubble expansion force, the bubble will be compressed. In the bubble compressing process, the elastic potential energy decreases, whereas the bubble pressure rises, and when the bubble pressure reaches a certain value, the bubble starts to expand again. The aforementioned process repeated under a certain conditions, bubble oscillation occurs. Considering the continuity and momentum equations as well as the force balance on bubble wall and the liquid boundary remote from the bubble wall, the compressed bubble inflation may be described as

$$\ddot{R} = \frac{P_g}{R_p} - \frac{2\sigma}{\rho R^2} - \frac{P_\infty}{\rho R} - \frac{3\dot{R}^2}{2R\rho} - \frac{12\eta_0}{R\rho\lambda_0} \int_0^t e^{-(s-t/\lambda)} \frac{R^2(s)\dot{R}(s)}{R^3(t)-R^3(s)} \ln \left| \frac{R(t)}{R(s)} \right| ds \quad (5)$$

where P_g is the gas pressure in the bubble. P_∞ is the pressure of polymer solution remote from the bubble surface. σ is the surface tension of the bubble. R is the bubble radius. η_0 is the zero-shear viscosity. ρ is the density of polymer solution. λ is the relaxation time of the polymer.

According to Fick's law of diffusion, the relationship between P_g and R can be shown as

$$P_g = \left[\frac{6\rho^2 D (C_0 - C_w)^2 R}{\rho_g R^3 - \rho_{g0} R_0^3} - \frac{3\dot{R}\rho_g}{R} \right] \frac{R_g T}{M} \quad (6)$$

where ρ_g is the density of gas in the bubble. ρ_{g0} is the initial density of the gas in the bubble. D is the diffusion coefficient. C_0 is the initial concentration of dissolved gas in the polymer solution. C_w is the concentration of dissolved gas in the polymer solution on the bubble surface wall. T is the temperature. M is the relative molecular mass of the gas. Using Eq. (1) and Eq. (2), we can get the bubble inflation process, shown as Fig. 2 ($P_g = 0.4052\text{MPa}$, $P_\infty = 0.1013\text{MPa}$, $\lambda = 0.9\text{s}$, $\rho = 0.89\text{g/cm}^3$, $\sigma = 2.97 \times 10^{-2}\text{N/m}$).

The bubble oscillatory frequency in Fig. 2 reached the order of 10^7Hz . In the very early period of bubble inflation, the initial bubble radius is small. Let the initial bubble radius equal $1\mu\text{m}$, the stretching rate reaches the order of 10^6s^{-1} .

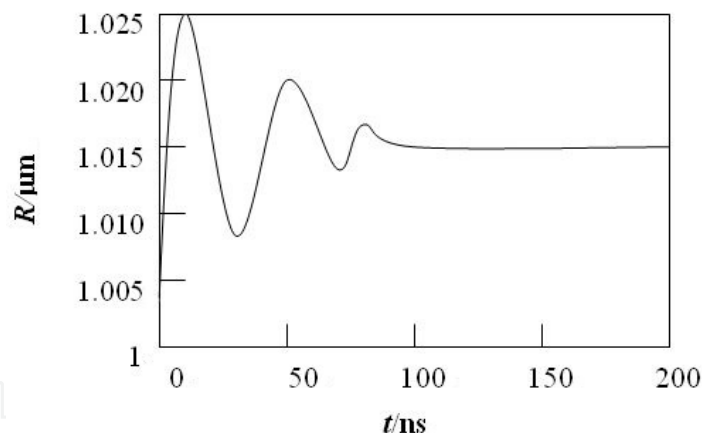


Fig. 2. Variation of bubble radius in the process of bubble inflation

According to Eq. (3), the minimum granule radius that can be separated under the bubble oscillation shown in Fig. 2 is 6 nm.

On the other hand, in the anaphase of diminutive bubble inflation, bubble instabilities may occur and eventually cumulative jet penetrate the bubbles, just as Fig. 3 shows. The cumulative jet leads to micro-droplets formation and subsequent evaporation. The micro-droplets are spatially homogeneously distributed inside the gas-nanomaterials mixture. The mixture process made nanomaterials in the bubbles enwrapped with liquid polymer micro-droplets and evaporation, which ameliorates the interphase binding state between nanomaterials and liquid polymer. What's more, the bubble oscillation frequency reached ultrasonic frequency, ultrasonic cavitations may appear. This can also promote nanoparticles dispersion.



Fig. 3. Cumulative jet

2.2 Preparation system

The polymer nanocomposites preparation system includes compressed gas preparation subsystem, gas-nanomaterials mixture subsystem and polymer nanocomposites preparation subsystem (shown as Fig. 4).

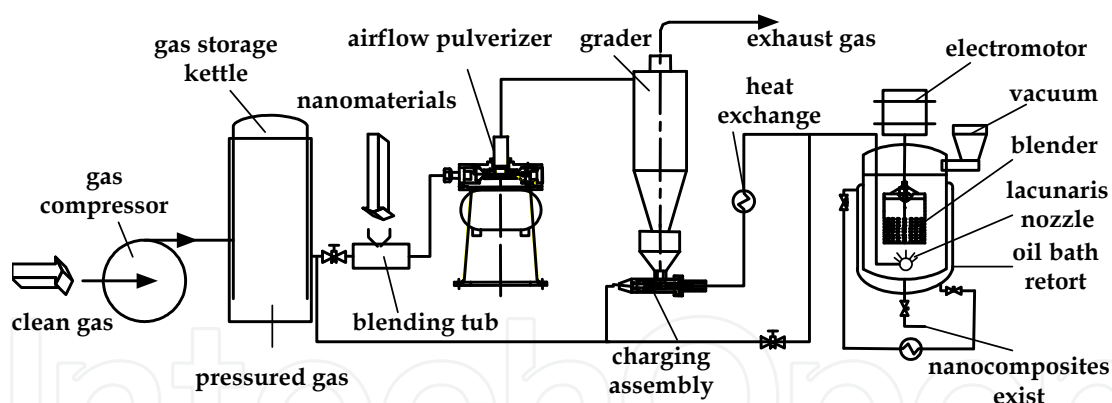


Fig. 4. Sketch of the nanocomposites preparation system

2.2.1 Compressed gas preparation subsystem

The compressed gas preparation subsystem is made of gas compressor, gas storage kettle and some accessories, which can supply compressed gas for nanocomposites preparation.

2.2.2 Gas2nanomaterials mixture subsystem

The gas-nanomaterials mixture subsystem is made of blending tube, siphon and heat exchanger. In the gas-nanomaterials mixture process, nanomaterials were commixed with compressed gas. That leads to gas-nanometer scale particles mixture jet formation. A blending tube was designed to commix nanomaterials into compressed gas (Fig. 5).

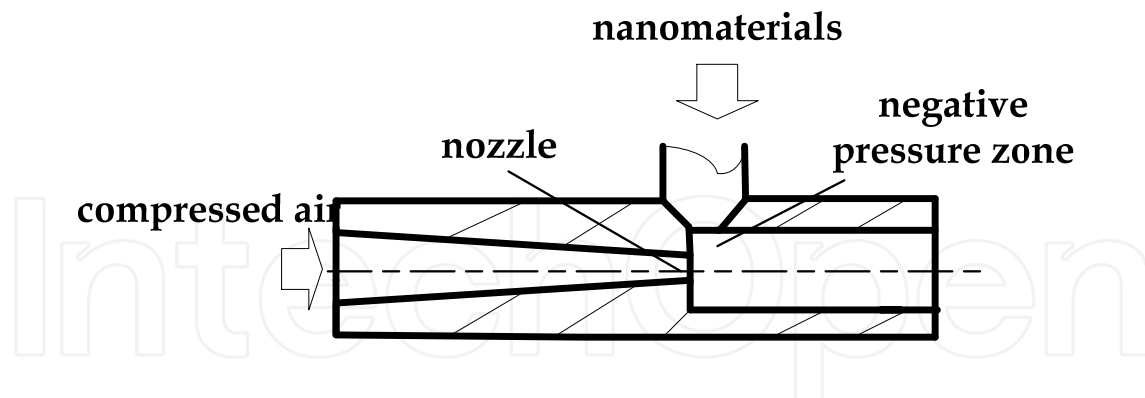


Fig. 5. Blending tube

The compressed gas from gas storage kettle turns into high-velocity jet when it rushes through the small nozzle in blending tube. Because of the high-velocity gas flow's entrainment effect, a negative pressure zone is formed around the nanomaterials entrance. As the pressure of negative zone is much lower than atmospheric pressure, SiO_2 powder will be absorbed into the blending tube, where the SiO_2 powder is blended with the gas flow by the way of momentum exchange and forms SiO_2 powder-gas admixture. When the admixture rushes through the siphon (whose inner diameter is 4 mm) between blending tube and lacunaris nozzle, the impact and shear caused by the high-velocity gas flow rubbing and impacting with pipe wall lead to SiO_2 powder conglomerations' being elementarily destroyed to smaller particles. The heat exchanger increases the temperature of the gas-nanomaterials so high that the liquid polymer near the lacunaris nozzle will not be cooled.

2.2.3 Polymer nanocomposites preparation subsystem

The polymer nanocomposite preparation subsystem is made of oil bath retort, cylinder blender, electromotor, vacuum pump and lacunaris nozzle. The oil bath retort makes the retort inner temperature high enough to keep polymer in liquid state. The gas-nanomaterials jet from lacunaris nozzle rushes into oil bath retort. As the gas-nanomaterials jet is an under-expanding jet, it will turn into a mass of diminutive bubbles distributing in the liquid. The diminutive bubbles with compressed gas and nanomaterials are closed inside working as nanomaterials dispersion energy cell and minisize nanometer scale particles storage cell.

The vacuum pump keeps the retort in low pressure. As the pressure in the bubbles is much higher than the retort inner pressure, bubble expansion occurs in the liquid. During the bubble expansion, the polymer solution around the bubble is subject to stretching with high stretching rate. The high stretching rate will disperse nanomaterials agglomeration granules effectively.

Cylinder blender includes a cylinder with a lot of holes and a parabola impeller. The high speed electromotor drives the impeller to rotate with high speed. When the impeller is rotating, there will be a pressure difference between the polymer solution in the cylinder and those out of it, just like impeller pump do. The difference of pressure drives the polymer solution to rush through the holes in the cylinder. That will do strong shear and stretching action on the admixture liquid. The impeller can drive the polymer solution

rotating at high speed. Inflated bubble may collapse in this process. The energy released by bubble collapse or explosion may disperse the nano-particles into polymer ulteriorly.

2.3 Experiment

Raw material : epoxy resin E-20, produced by XIN GCHEN chemical new material Co. Ltd. Intenerating point: 71°C, the epoxy value: 0.20ep/100g, hydrolyzed hydrogen: 0.03%, volatile: 0.3%. Solidifying reagent: T31. A. R. acetone, produced by Harbin XINCHUN Chemist Plant.

Nanomaterials: nanometer SiO₂ (the diameter is about 20 nm). The nanometer SiO₂ is shown as Fig. 6.

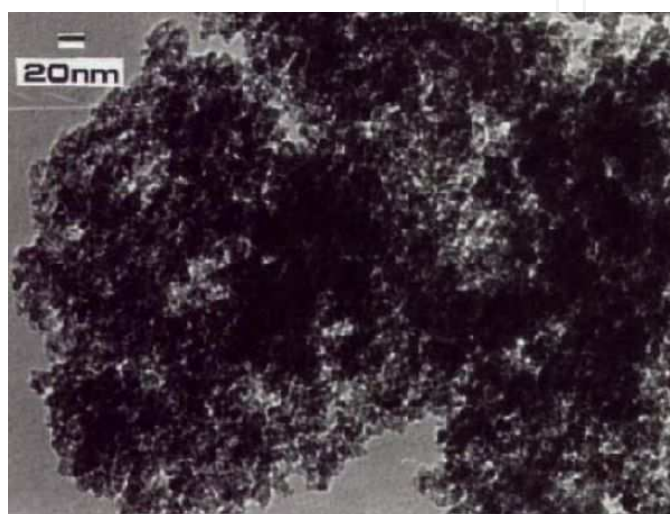


Fig. 6. Nano-SiO₂ TEM image

Dissolving 500g epoxy resin into 1500mL A. R. acetone, then we configure them into solution. The epoxy resin/acetone solution's density (denoted as ρ) is 0.93g/cm³, moving viscosity at 20°C(denoted as ν) is 7.75×10^{-6} m²/s, and dynamical viscosity (denoted as μ) is 7.21×10^{-3} Pa · s, surface tension (denoted as σ) is 2.97×10^{-2} N/m. The contact angle of epoxy resin/acetone solution and SiO₂ at room temperature is measured by contact angle analyzer, which is less than 90°. The epoxy resin/acetone solution and SiO₂ is soakage. That's to say, the surface energy of SiO₂ and epoxy resin/acetone solution is almost at the same order. As the epoxy resin/acetone solution and SiO₂ is soakage, the SiO₂ particles will accrete on the bubble wall during the process of bubble expansion and raising. The energy released by the bubbles explosion will disperse the nanometer scale particles into liquid polymer.

The pressure of compressed gas is 0.4MPa, the flux of pressured air (denoted with Q) is 44 L/min. The diameter of lacunaris nozzle is 4mm. We spout 10g nanometer SiO₂ into the solution from the jet gun. The epoxy resin/acetone solution is milled at the speed of 3000 r/min. After spouting, we make it mill for 20min, then put away the admixture of epoxy resin and SiO₂ for 12h. Then, we take out 20mL of the admixture of epoxy resin and SiO₂. By adding 5mL T31 into the 20mL solution, it will solidify. When epoxy is solidified, we make the sample slice from it and observe it under TEM.

According to Eqs. (3), (5), (6), the minimum granule radius that can be separate under the bubble oscillation under the condition $P_g=0.4052$ MPa, $P_\infty=0.1013$ MPa, $\lambda=0.9$ s, $\rho=0.89$ g/cm³, $\sigma=2.97 \times 10^{-2}$ N/m is about 6 nm. Then, the SiO₂ may be dispersed into epoxy as the configuration of nanometer scale particles.

2.4 Results and discussion

Effective distribution and dispersion is necessary for the preparation of polymer/nano-particles composites. The diminutive bubbles explosion dispersion method is a dispersion mixing method and has significant effect on dispersion agglomerated nano-particles. A CM-12 transmission electron microscope (made by Philip Co. Ltd) was used to observe the E-20/SiO₂ nanocomposites prepared by the proposed method. The TEM image was shown as Fig. 7. The TEM images show the nano-SiO₂ equably dispersed in polymer by the configuration of 15nm-30nm particles.

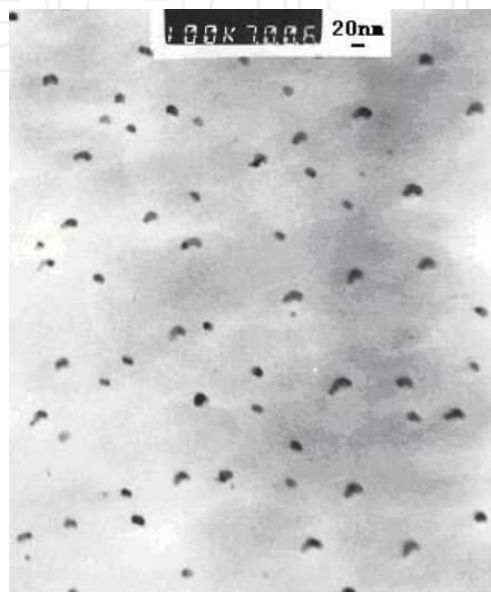


Fig. 7. E-20/ SiO₂ nanocomposites TEM image

Compared to traditional dispersion methods, the diminutive bubble explosion dispersion method is more effective in dispersion of nano-particles aggregates in polymer without dispersant because the particles aggregates serve as nucleation agents, and the bubbles expand on the surface of the aggregates. During the bubble inflation, the polymer surrounding the inflated bubble is subject to stretch in both longitudinal direction and latitudinal direction over the surface of inflated bubble. The bidirectional stretching increases the freedom degrees of nano-particles dispersion, which favors more homogeneous dispersion. The diminutive bubble explosion dispersion method may obtain ultrahigh stretching rates of around $(10^5-10^6)s^{-1}$, which is superior to the shearing dispersion effects obtained with screw extruders that have shearing rate about $(10^3-10^4)s^{-1}$.

According to the numerical simulation, the initial radii of diminutive bubbles have close relation to the stretch rate and bubble oscillation frequency in bubble inflation. The smaller initial radii of the bubble will lead to the higher stretch rate and oscillation frequency. High stretch rate and oscillation frequency favor more microcosmic homogeneous dispersion.

3. Nanocomposites capability evaluation method base on fractal theory and TEM image

Size distribution and space distribution of dispersed phase in nanocomposites is one of the key factors that effect material performance, and it's also an important indicator for evaluating the effect of nano-particles dispersion.

Size distribution of dispersed phase in nanocomposites means the range of particle diameter dimension. Size distribution of dispersed phase reflected the broken up effect of the agglomerated nanomaterials granules. The wider the range of size distribution of dispersed phase is, the worse the broken up effect of the agglomerated nanomaterials granules is. Wider range means that different degree of dispersed phase agglomeration phenomena exist. On the contrary, the narrower the range of size distribution of dispersed phase is, and the size accords with the demands of required nanometer scale, the better the broken up effect of the agglomerated nanomaterials granules is. Narrower range means that dispersed phase agglomeration is light.

Space distribution of dispersed phase in nanocomposites means the uniformity of nanoparticles dispersed into polymer and the uniformity of the number of nano-particles in unit volume polymer. Symmetrical dispersed phase can make the performance of nanocomposites good consistency. Contrarily, asymmetrical dispersed phase will result in stress concentration of nanocomposites and the performance of nanocomposites bad consistency.

3.1 Digital processing of nanocomposites TEM images

Because nanocomposites TEM images usually have the characteristic that their background brightness isn't homogeneous and gray scale difference between dispersed phase boundary and background is very small, we need to do some digital processing to TEM image for extracting the number of image elements in dispersed phase.

3.1.1 Balancing partition gray scale

Divide TEM image into $n \times n$ grids. Then, calculate gray scale of every image element according to Eq. (7) for balancing gray scale of the whole image whose gray scale average value will turn into 128.

$$g_{n1} = g_a - (m_1 - 128) \quad (7)$$

where g_{n1} is new gray scale of a image element. g_a is original gray scale of that image element. m_1 is gray scale average value of the grid which the image element belong to.

3.1.2 Adjusting brightness of every image element

Make every image element as a center. Choose a grid include $k \times k$ image elements. Then, calculate gray scale value of every image element according to Eq. (8) for adjusting brightness so that every image element in the whole image will has almost the same brightness.

$$g_{n2} = g_a - (m_2 - 128) \quad (8)$$

where g_{n2} is new gray scale of a image element. g_a is original gray scale of that image element. m_2 is gray scale average value of the grid whose center is the image element.

3.1.3 Superimposing image

Superimpose the two images which received separately from the two steps aforementioned according to Eq. (9), for increasing gray scale difference between dispersed phase and background.

$$g_{n3} = g_{n1} - (g_{n2} - 255) \quad (9)$$

where g_{n3} is gray scale of a image element after superimposing. If $g_{n3} < 0$, set $g_{n3} = 0$.

3.1.4 Thresholding

After the processing aforementioned, transform the image into binary image through assigning gray value 0 to image elements that belong to dispersed phase and gray value 255 to other image elements.

3.2 Theory of sand box method and gyration radius method

We can measure the number of particles in a ball with its radius ε . If particles are distributed on a plane, the number $M(\varepsilon)$ of particles on the cross section of a ball (radius ε) can be expressed as $M(\varepsilon) \propto \varepsilon^2$. If particles are distributed in space in a ball (radius ε), the number $M(\varepsilon)$ of particles can be expressed as $M(\varepsilon) \propto \varepsilon^3$. In a similar way, if the distribution of particles has the characteristic of self-similarity and the fractal dimension is D , we can get the expression that

$$M(\varepsilon) \propto \varepsilon^D \quad (10)$$

where $M(\varepsilon)$ is the number of dispersed phase particles in a box (radius ε). D is fractal dimension. Here, we finish the definition of sand box method. On the base of sand box method theory, we can continue to derive gyration radius method.

Define gyration radius R_g , it can be expressed as

$$R_g(Z) = \left(\frac{1}{Z} \sum_{i=1}^Z r_i^2 \right)^{\frac{1}{2}} \quad (11)$$

where r_i is the distance between particle i and the center of fractal mass. Z is the number of particles in the box whose center is O and radius is R_g .

According to Eq. (10), if make R_g as the radius of a box, the number of particles in the box can be expressed as

$$Z \propto R_g^D \quad (12)$$

Eq. (12) can be transformed into another form that

$$R_g \propto Z^{\frac{1}{D}} \quad (13)$$

3.3 Size distribution of dispersed phase in nanocomposites

After transform TEM image into binary image, we need to calculate the number of image elements in every dispersed phase and make eight neighbourhoods mark to every dispersed phase in binary image. The number of image elements with the same value mark is defined as the area (S_k) of a corresponding dispersed phase (k). Then, the equivalent radius (R_k) can be expressed as

$$R_k = \sqrt{\frac{S_k}{\pi}} \quad (14)$$

Determine the centers of dispersed phases according to centroid method. Then, mark every center with the equivalent radius value of corresponding dispersed phase and other image elements with 0. Then, the TEM image turned into dot matrix image. The dot matrix image can be reduced to a row matrix, in which the first element is 0 and the second to the $h+1$ elements are the equivalent radius values arranged from small to small. h is the size fraction of disperse phase equivalent radius. The first element is defined as the center of the row matrix.

The dispersed phases in nanocomposites can be regarded as fractals making up of dispersed objects and have the characteristic of self-similarity. According to Eq. (10), the size function of dispersed phase can be expressed as

$$Q(r) \propto r^p \quad (15)$$

where $Q(r)$ is the number of dispersed phase in a circle whose radius value is r . p is the parameter of size distribution of dispersed phase.

Then, the number of elements (N_1) that the distances between the center of the row matrix and them are smaller than step-length (m) can be given by

$$N_1 = K_1 m^p \quad (16)$$

where K_1 is constant.

Set different step-length ($m=1, 2, 3\dots$), calculate N_1 according to Eq. (15), then we can get a series of datum ($\ln(m), \ln(N_1)$), in which $\ln(\)$ is natural logarithm. Then, we can transform Eq. (16) to another form that

$$\ln(N_1) = p \ln(m) + \ln(K_1) \quad (17)$$

Obviously, we can draw a line with the points ($\ln(m), \ln(N_1)$) in double-log coordinate. The linear slope is just the parameter p . Because K_1 is a constant, $\ln(K_1)$ can not affects slope p .

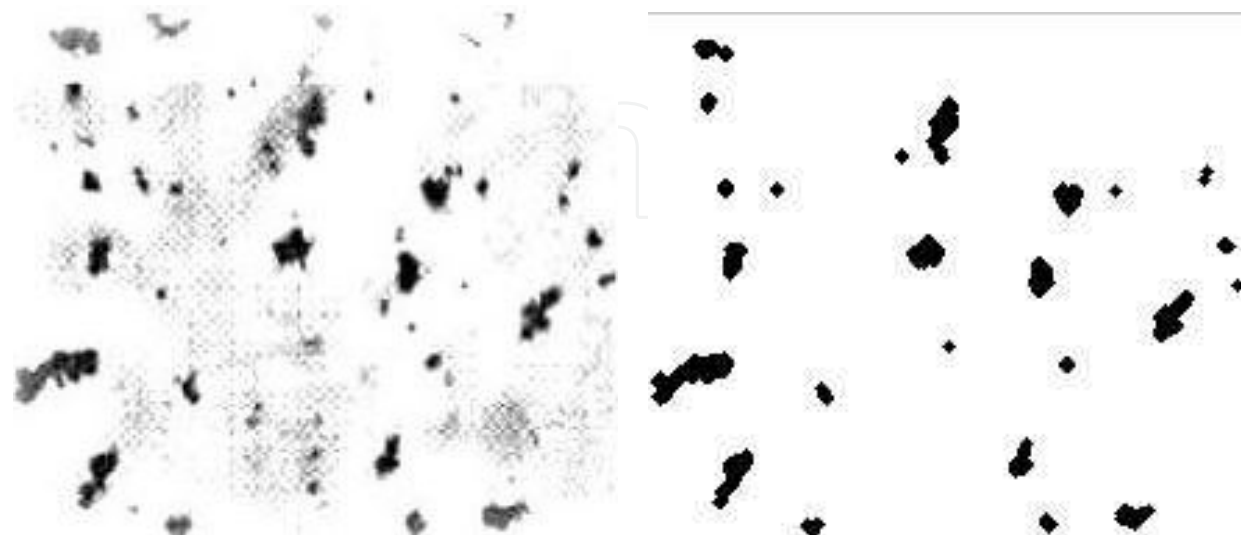


Fig. 8. Nano-composites SiO_2 / acrylic esters by shearin(TEM image(left), binary image (right))

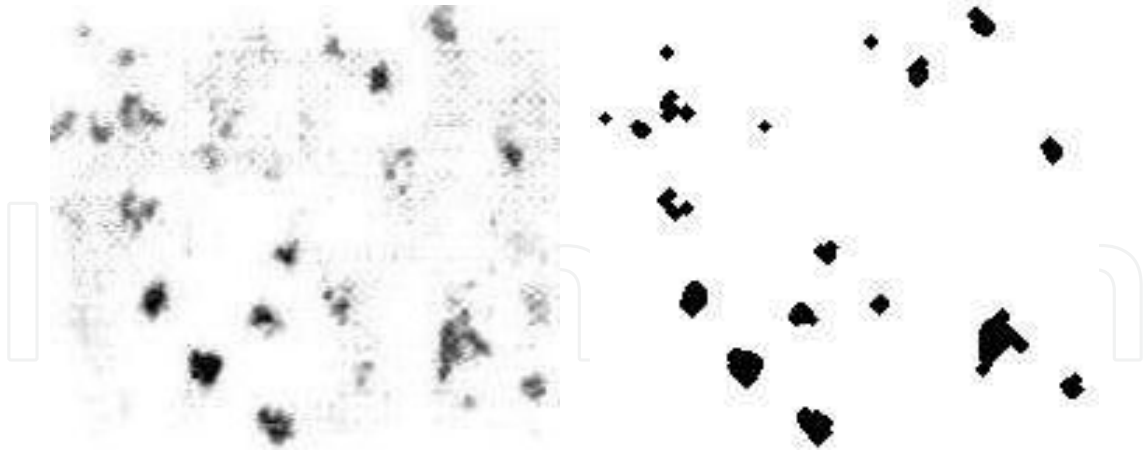


Fig. 9. Nano-composites SiO_2 / acrylic esters by high speed ball milling(TEM image(left), binary image(right))

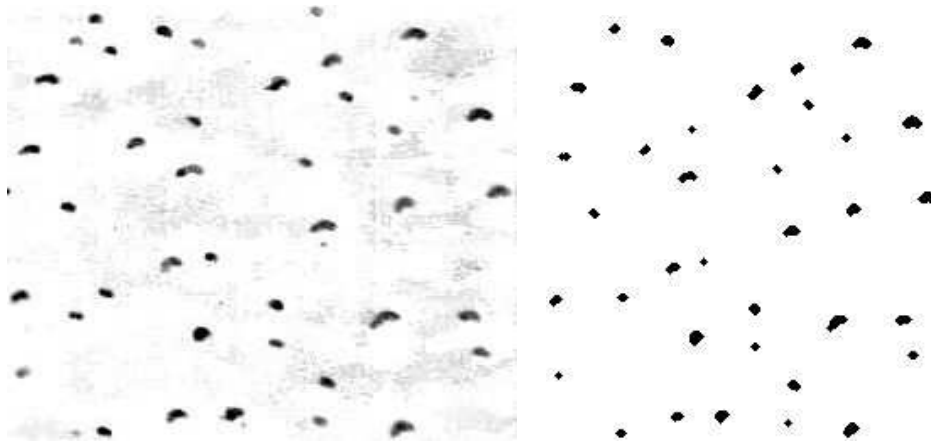


Fig. 10. E-20/ SiO_2 nanocomposites by inflated bubble(TEM image(left), binary image(right))

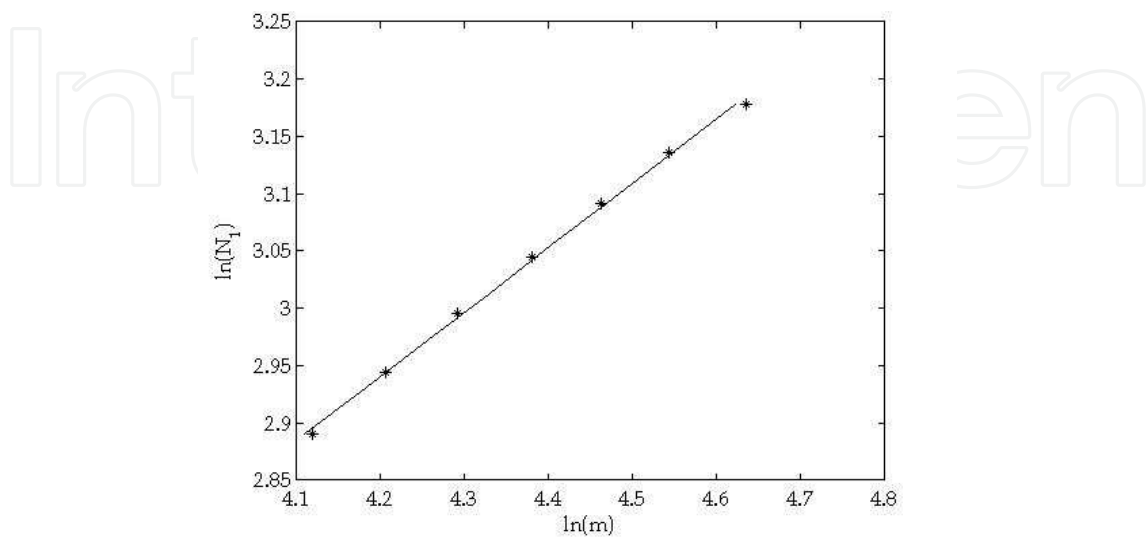


Fig. 11. Fitting curve of figure 8 with dimension p_1

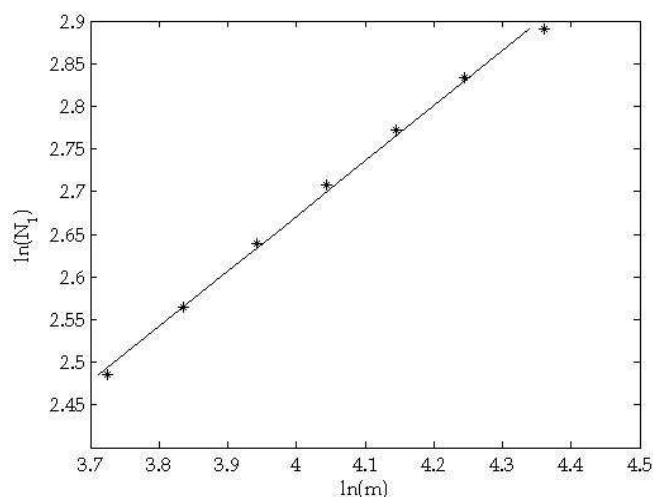


Fig. 12. Fitting curve of figure 9 with dimension p_2

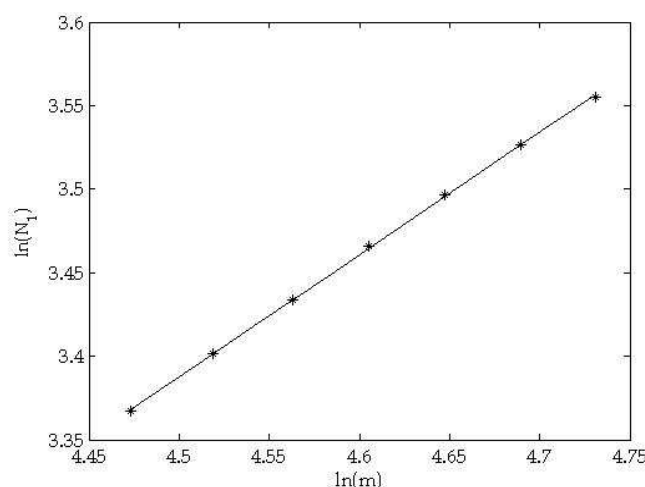


Fig. 13. Fitting curve of figure 10 with dimension p_3

Fig. 8, 9, 10 are TEM images and binary images of three kinds of composite. Fig. 11, 12, 13 are fitting curves correspondingly drawn according to Eq. (17). p_1 , p_2 , p_3 are their slopes as well as parameters of size distribution of dispersed phase. After calculating, we can know that $p_1=1.806$, $p_2=1.793$, $p_3=1.382$ and $p_1 > p_2 > p_3$. According to Fig. 8, 9, 10, we can easily discover that their size distribution of dispersed phase status has relatively obvious difference. In Fig. 8, dispersed phase agglomeration phenomena is the most serious, and the range of size distribution is the widest. In Fig. 10, dispersed phase agglomeration phenomena is the slightest, and the range of size distribution is the narrowest. It can be shown that the smaller the value of p is, the narrower the range of size distribution is, and parameter p can be used to describe the fractal characteristic of size distribution of dispersed phase.

3.4 Space distribution of dispersed phase in nanocomposites

After transform TEM image into binary image, determine the center of TEM image and the centers of dispersed phases according to centroid method. Then, mark every centers of dispersed phases with 1 and other image elements with 0. Then, the TEM image turned into dot matrix image.

According to Eq. (11), we can get the equation that

$$R_g = \left(\frac{1}{N_2} \sum_{i=1}^{N_2} r_i^2 \right)^{\frac{1}{2}} \quad (18)$$

where r_i is the distance between the two centers of dispersed phase i and TEM image. N_2 is the number of dispersed phases in the circle whose center is O and radius is R_g .

According to Eq. (13), the space distribution uniformity function of dispersed phase can be expressed as

$$R_g \propto N_2^{\frac{1}{q}} \quad (19)$$

where q is the parameter of space distribution uniformity of dispersed phase.

Set a constant K_2 , we can get Eq. (20) that

$$R_g = K_2 N_2^{\frac{1}{q}} \quad (20)$$

Draw a circle with the center O and the radius m_i ($i=1, 2, 3, \dots$), then calculate the number (N_2) of dispersed phase and radius r_i corresponding to every dispersed phase in the circle. Then, we can calculate R_g according to Eq. (18). Choose different radius m_i , we can get a series of datum ($\ln(N_2), \ln(R_g)$).

Eq. (20) can be transformed to another form that

$$\ln(R_g) = \frac{1}{q} \ln(N_2) + \ln(K_2) \quad (21)$$

Obviously, we can draw a line with the points ($\ln(N_2), \ln(R_g)$) in double-log coordinate. The reciprocal of the linear slope is just the parameter q . Because K_2 is a constant, $\ln(K_2)$ can not affect slope parameter q .

Fig. 14, 15, 16 are fitting curves correspondingly drawn according to Eq. (17) corresponding to Fig. 8, 9, 10 respectively. q_1, q_2, q_3 are the reciprocal values of the linear slopes as well as parameters of space distribution uniformity of dispersed phase respectively. After calculating, we can know that $q_1=1.3604, q_2=1.535, q_3=1.991$ and $q_1 < q_2 < q_3$. According to Fig. 8, 9, 10, we can easily discover that their space distribution of dispersed phase status has relatively obvious difference. In Fig. 8, the distribution uniformity is the worst. In Fig. 10, the distribution uniformity is the best. Our research shown that the ideal fractal dimension is 2 for the fractal whose particle are dispersed on a plane. It can be shown that the closer the value of q is to 2, the better the the distribution uniformity is, and parameter q can be used to described the fractal characteristic of space distribution uniformity of dispersed phase.

4. Conclusion

The epoxy resin/SiO₂ nanocomposites preparation method based on diminutive bubbles explosion can evenly disperse SiO₂ particles into epoxy resin as the configuration of nanoscale particles. At the very start of bubble explosion, the bubble will expand and lead to high stretching rate in the polymer surrounding the bubble. The stretching rate may reach 10⁶s⁻¹, which favors more homogeneous dispersion of nano-particles. Diminutive bubbles explosion method can be used for the dispersion of nanometer scale particles.

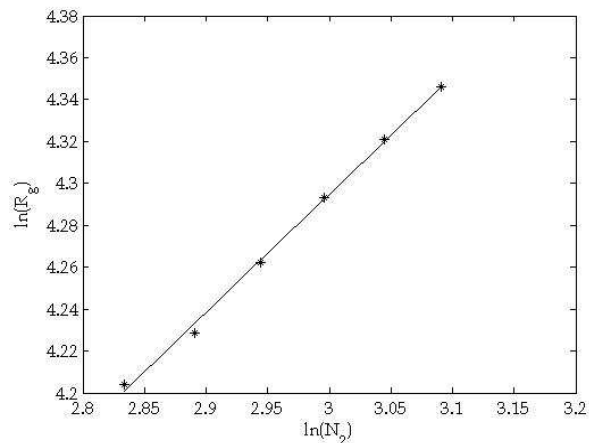


Fig. 14. Fitting curve of figure 8 with dimension q_1

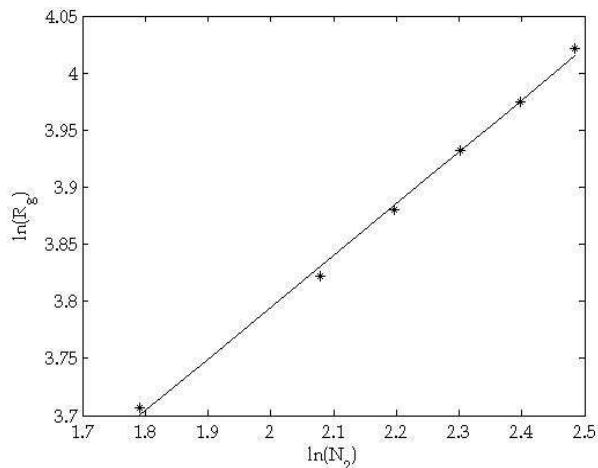


Fig. 15. Fitting curve of figure 9 with dimension q_2

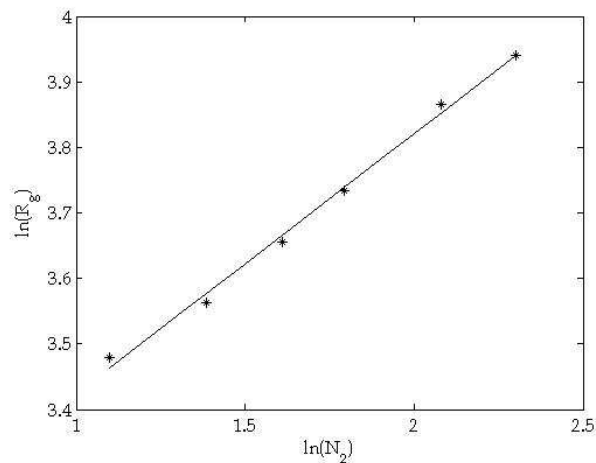


Fig. 16. Fitting curve of figure 10 with dimension q_3

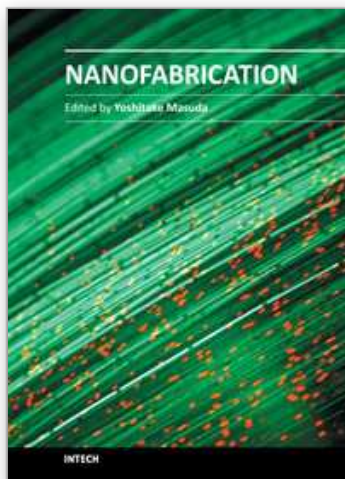
A nanocomposites preparation system was designed. By using the preparation system, epoxy resin/SiO₂ nanocomposites were prepared. The SiO₂ dispersed into epoxy as the configuration of 15nm - 30nm particles.

A quantitative description method was proposed to evaluate size distribution and space distribution of dispersed phase in nanocomposites based on fractal theory and TEM image

of nanocomposites. The models of size distribution and space distribution of dispersed phase were established using fractal dimension of natural fractal. After TEM image processing, parameters of size distribution and space distribution uniformity of dispersed phase were extracted. Then, lines were fitted according to linear equations correspondingly and it is obvious that the parameters and the values of linear slope have corresponding relations. Using the parameters to describe distribution of dispersed particles in the nanocomposites, the results show that it can exactly evaluate the size distribution and space distribution of nano-particles.

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