

EPOXY-SILICA NANOCOMPOSITE: CREEP RESISTANCE AND TOUGHENING MECHANISMS

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Abstract

In this article, the effect of modified nanosilica (m-nanosilica) on the mechanical and creep performance of epoxy resin were investigated. At the same time, the toughening mechanisms of epoxy resin with m-nanosilica were also considered. The results show that the crack pinning and crack deflection mechanism contributes only a small part to nanocomposite, this due to the size of the nanosilica particles is such smaller than crack-opening displacement. The FE-SEM surface fracture shows evidence of the debonding of the nanoparticles from the polymer matrix and the expansion of the holes around the nanoparticles as well as the existence of the plastic deformation zone. Plastic deformation and plastic void growth are the main toughening mechanism in nanocomposite material.

Keywords: Epoxy; nanosilica; KR-12; creep and recover; toughness mechanism.

1. Introduction

For the conventional polymer composite, one of the weakest points is the bonding between the reinforcing layers, which limits their application in practical. Therefore, it is essential to improve the strength of substrate phase to fabricate the high-quality composite. In recent years, nanotechnology is emerging as a promising tool to maximize the material properties. In order to completely employ the potential of the nanotechnology, it is necessary to have a suitable simulating model allowing prediction of macro mechanical properties from the material structure and the effects of nano phases to fabricate the most suitable material for specialized applications. These models allow to explain the drain of majority energy based on the different destructive mechanisms of the nano materials. Nanocomposites are the composite of the substrate phases, which are reinforced with suitable nano particles, so that the nanocomposite properties such as hardness, tensile strength, abrasive resistance and toughness strength... are significantly improved with nano additives contents compared to initial polymers from average to insignificant amount.

Epoxy resin, one of the most popular used thermoset plastics in the globe, has the following advantages such as high adhesion on many various substrates, relatively high

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mechanical strength and hardness. However, epoxy has disadvantage of brittle, low toughness lead to distruction of materials when crack. There have been extensive works on purpose of increasing the toughness of epoxy by combination with rubber powder [1] or other thermoplastics [2, 3], however beside the enhancement of toughness, other properties such as mechanical, module and glass transision temperature are significantly affected. Researchers so far demonstrated that the use of nanoparticles including silica, clay, carbon, graphene, metal oxides... was a promising pathway to improve toughness of epoxy without affecting to initial properties of materials [4-6]. Currently, the destructive mechanism in nanocomposite is extensively studied by scientists all over the world. Several models of toughing mechanism of epoxy in the presence of reinforcing nanoparticles have been reported such as the crack path deflection, crack pinning, plastic deformation, leaking of nanoparticles out of polymer substrates and crack void growth... In this study, nanocomposite is fabricated from expoxy resin and nanosilica modified by KR-12 titanate coupling agent. The effects of each destructive mechanism on the enhancement of mechaniscal strength as well as creep resistance of nanocomposite are investigated in detail.

2. Experiment

2.1. Materials

Bis-phenol A epoxy resin (DGEBA type YD-128) with an epoxy equivalent weigh of 188 g/mol was purchased from Dow Chemical Company; Titanate coupling agent KR-12 (isopropyltri (dioctylphosphate) titanate provided by Ken-React Petrochemicals, Inc); tetrabutyltitanate (TBuT 99.0%, $C_{16}H_{26}O_4Ti$, MW 340.32 g/mol) as a hardener was supplied by Sigma Aldrich. Nanosilica K-200 (Korea) purchased from DC Chemical Co., Ltd., average particles size of 20 - 30 nm, specific surface area of 200 m²/g was used for preparation of epoxy/modified silica (m-silica) nanocomposites. Acetone (AR grade, China 99.5%) was used as purchased without any treatment or purification.

2.2. Preparation of epoxy/modified silica composites

Surface of silica nanoparticles was treated by KR-12 titanate coupling agent according to [7]. To prepare epoxy/modified nanosilica (m-nanosilica) composites, a series of various m-nanosilica content dispersed in acetone was mixed with epoxy resin (EP) on a mechanical stirring device at a speed of 1500 rpm for 30 minutes and ultrasonic treatment at a speed of 20000 rpm for 1 hour at room temperature. Then, the mixture was heated under a vacuum oven at 65°C for 5 hours to remove acetone solvent. Afterwards, the TBuT hardener was added into above mixture (the weight mixing ratio of the epoxy and the hardener is 100:15). The mixture with hardener was continuously stirred and

ultrasonicated until obtaining a uniform mixture. Next, the mixture was degassed by vacuum-pumping for 1 hour at room temperature. Finally, the mixture was poured into a polytetrafluoroethylene used mold. The samples were cured in an oven at 120°C for 3 hours. An epoxy density of 1.1 g/cm³ and silica density of 2.2 g/cm³.

2.3. Characterization and measurements

2.3.1. Mechanical properties

A tensile strength of the epoxy/modified nanosilica (m- nanosilica) composites was measured by a universal testing machine (INSTRON 5582-100KN, USA) in accordance with the standard ASTM D638. The rate of tensile deformation was 1 mm/min. Young's modulus was determined from the strain and tensile load. The values of the Young's modulus were obtained by measuring five times and then averaging.

2.3.2. Microscopic analysis

Field emission Scanning electron microscopic (FE-SEM) analysis of the epoxy/m-nanosilica composites was done to investigate the debonding of nanosilica in epoxy resin, plastic deformation and plastic void growth of polymer, examined using HITACHI S-4800 instrument (JEOL, Japan) at an accelerating voltage 15 kV.

2.3.3. Fracture toughness and fracture energy

Fracture toughness of the epoxy/m- nanosilica composites was determined using a LLoyd 500 N (England) material testing instrument, following the ASTM standard D5045 as shown in Figure 1. The plane strain fracture toughness (K_{IC}) was measured using pre-cracked, single edge notched, specimens in three-point bending geometry. A pre-crack was made by lightly tapping a fresh razor blade between adjoining plates, with samples dimension of 120 mm (length) × 12.0 mm (width) × 6.0 mm (thickness). For each composite, 5 specimens were evaluated at a rate of 1 mm/min. The fracture toughness of sample was calculated using the following (1) and (2) equations where P_Q is maximum load, B is width of the specimen; a is crack length; W is height of the specimen; $x = a/W$.

$$K_{IC} = \left(\frac{P_Q}{BW^{1/2}} \right) f(x) \quad (1)$$

$$f(x) = 6x^{1/2} \frac{[1.99 - x(1-x)(2.15 - 3.93x + 2.7x^2)]}{(1+2x)(1-x)^{3/2}} \quad (2)$$

$$0.45 \leq a/W \leq 0.55.$$

The fracture energy (G_{IC}) of sample was calculated from K_{IC} and E , using the relationship:

$$G_{IC} = K_{IC}^2 [(1 - \mu^2)] / E \quad (3)$$

where E is the modulus of elasticity of sample was obtained from tensile test, μ is the Poisson's ration.

$$\mu = - \frac{\varepsilon_{trans}}{\varepsilon_{axial}} \quad (4)$$

where ε_{trans} is transverse strain, ε_{axial} is axial strain.

2.3.4. Creep and recover test

Tensile creep and recover test of the epoxy/m- nanosilica composites was measured by a universal material tester (WP-300, Germany) in accordance with the standard ASTM 2990-01. Two strain gauge extensometer with span lengths of 40mm were used for strain measuring within accuracy of 0.005%. Three stress levels were chosen 5 MPa, 10 MPa and 15 MPa. Duration of the test 1200s creep and 1500s creep-recover. The values of the strain were obtained by measuring three times and then averaging.

3. Results and discussion

3.1. Creep resistance

Creep resistance is one of the most important properties of polymer, which is decisive factor on applicability of materials in practical. Unlike brittle destruction, creep failure is not suddenly occurred but experience an accumulation under stress over a long period of time. Creep and recovery capability of epoxy and nanocomposite at temeprature of 30°C is shown in Fig. 1. It is obvious that at the certain stress, deformation of samples increases along with time and the most deformed stage observed is within first 1200 seconds, the samples then are in stable stage. The pristine epoxy sample has larger deformation level in comparison with the samples reinforced by 33% nanosilca. The deformation level of nanocomposite reinforced with 1 - 5% nanosilia are virtually unchanged, however with nanosilca content of higher than 7% the deformation tends to increase, when impact stress increases from 5 to 15 MPa, the deformation of nanocomposite samples is also linearly increased. When remove the pressure, the samples quickly recover to the initial state in which the nanocomposite samples virtually reach to the initial diamters. The low deformation and quick recovery of the nanocomposite samples demonstrate that the use of nanosilica has benefit of improving the creep resistance for epoxy and changing the material from brittle to the toughing state in the same time, which help the epoxy-based nanocomposite expanding their applications in practical.

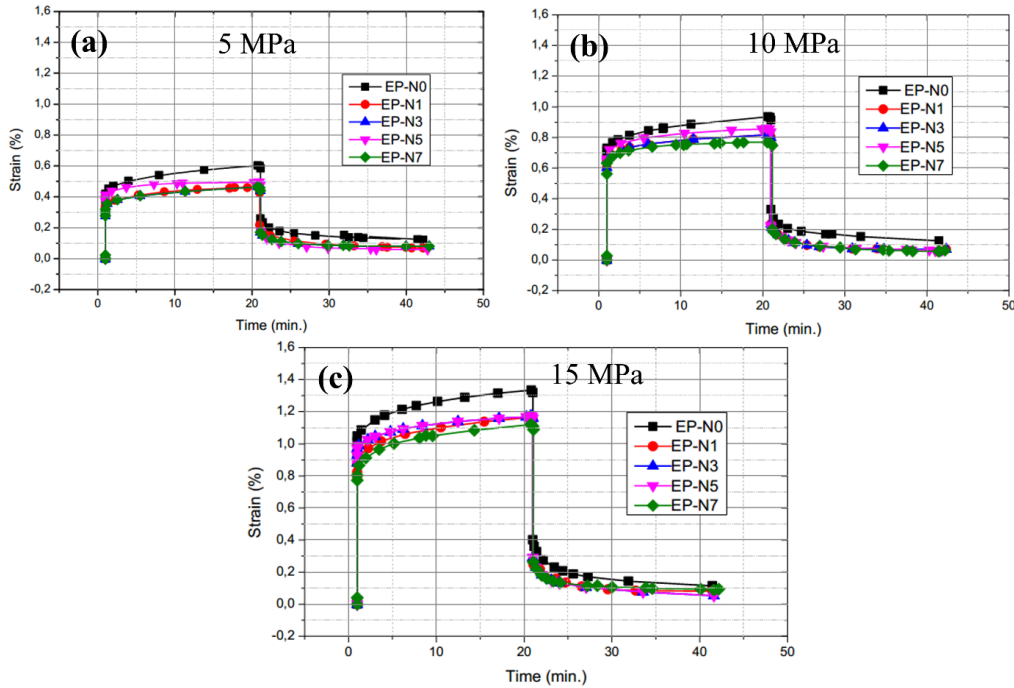


Fig. 1. Creep resistance and recovery capability of pristine epoxy and nanocomposites with various nanosilica contents at temperature of 30°C and at the stress of 5 MPa, 10 MPa, 15 MPa.

The enhancement of the creep resistance is ascribed to the rigid structure of nanosilica, which is homogeneously and interchangedly distributed in the epoxy substrate, generates a high surface area preventing the movement of polymolecules, control the slippery between layers and deformation of the substrate under external forces, lead to enhancement of the creep resistance of nanocomposites compared to the initial epoxy [8].

3.2. Toughness mechanisms

The effects of modified nanosilica on the mechanical strength, focusing coefficient of breaking stress, poisson coefficient... of the epoxy/nanosilica composite have been studies in our previous works [9] results are shown in following Table 1.

Table 1. Mechanical properties and fracture toughness of neat epoxy and nanocomposite

Samples name	Nanosilica content, wt%	Nanosilica content, vol%	Tensile strength, MPa	E_{tensile} , GPa	K_{IC} (MPa.m ^{1/2})	μ	G_{IC} (kJ/m ²)
EP-N0	0	0	57.3	3.45	0.97	0.330	0.243
EP-N1	1	0.53	68.74	3.75	1.27	0.358	0.375
EP-N3	3	1.56	70.13	3.82	1.55	0.363	0.546
EP-N5	5	2.63	76.3	3.93	1.73	0.365	0.66
EP-N7	7	3.72	63.84	3.66	1.45	0.383	0.47

The enhanced strength mechanism or the large drain of energy with the presence of reinforcing nano particles in polymer have been mentioned in the previous studies [9-11]. These include crack path deflection, crack pinning, plastic deformation and plastic void growth. Depending on types of plastic and reinforcing additives, these mechanisms impact on strength differently and effect of each mechanism on the total strength also varies. Some of these mechanisms are discussed in the following sections.

3.2.1. Crack deflection

The theory on the crack deflection was introduced by Faber and Evans [12, 13]. In this theory, when cracks appear in the material and contact with reinforcing particles, they will be forced to deflect, even twist out of initial crack-containing planar and further develop. This leads to increase the destructive surface area. Moreover, the crack deflection also causes the change in the stress state from the pulling form in the plane (mode I) to the hybrid form such as pulling and slippery state (mode I/II) in the circumstance of crossing cracks or pulling and twisting (mode I/III) in the case of the twisting cracks. In order to develop the aforementioned hybrid cracks, it is necessary to have a higher external force compared to that of the crack in mode I, which lead to the increase of the destructive surface area, as a result the toughness of the material improves. The work [14] indicated the dependence between enhanced destructive energy of materials (ΔG) under effect of the crack deflection follows the equation:

$$\Delta G = \frac{3\gamma_m V_{fp}}{2} \quad (5)$$

where V_{fp} is the volume of reinforcing particles in the materials, γ_m is the specifically destructive energy of substrate phase. In the study reported by Moloney [15] about nanocomposite based on epoxy and reinforcing particles, the γ_m value of nanocomposite was equal a half of the G_{IC} value of epoxy substrate. The deformed energy-releasing speed G_{IC} of epoxy substrate after aging in this study was calculated to be 243 J/m².

Based on equation (5), the ΔG value is determined from the deflection mechanism of the cracks upon reinforcing nanosilica contents, results are presented in Fig. 2. It is obvious from the Fig. 2 that the ΔG value linearly increase along with the increase of nanosilica contents, which correspond to surface roughness, the highest ΔG value of 13.57 J/m² observed at the nanosilica content of 7%. However, in comparison with the corresponding destructive energy, the energy contributed for the crack deflection is relatively small, which is around from 0.5 to 2.88%. This indicates that in the toughening mechanism of epoxy using nanosilica, the crack deflection only contributes partially, not

decisively, to the tilt and twist of the crack tip because of very small size of nanoparticles comparing to the micron scale.

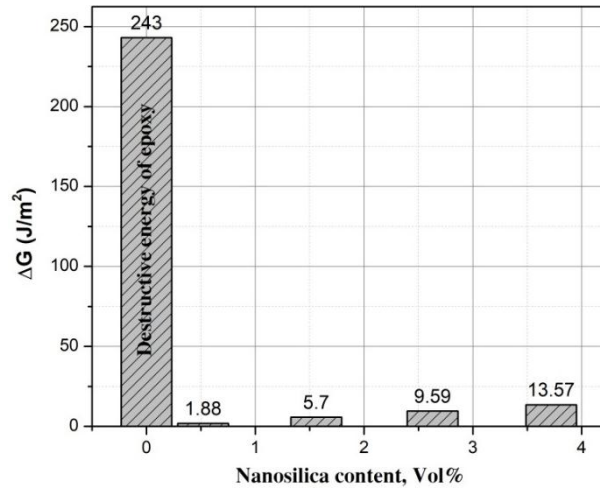


Fig. 2. Destructive energy difference of epoxy resin with and without einforcing nanosiclica.

3.2.2. Crack pining mechanism

Theory on the crack pinning mechanism was first introduced by Lange in 1970 [16] and further developed by Evan [17] and Green [18]. Based on these theories, when the cracks start spreading in the materials contact with reinforcing particles with high stiffness and good bonding with substrates, these cracks do not have enough energy to cut down or break the particles with substrates, therefore they tend to bend over the spaces between particles, lead to generate the sub-cracks, and after go through these particles these sub-cracks are reunited. This theory was used to explain for the toughing of the material upon addition of micron-size particles. Based on the theory, the crack pinning occurs when the size of the reinforcing particles is larger then the crack openness. In the planar deformation condition, plasticity area in front of the crack peak is silmulated as a plain area and the crack openness (δ_c) is determined as following equation:

$$\delta_c = \frac{K_{IC}^2}{E \cdot \sigma_c} (1 - \mu^2) = \frac{G_{IC}}{\sigma_c} \quad (6)$$

where σ_c is the elasticity limit of the substrate, G_{IC} is the destructive energy as determined by the documents [4, 7, 9] . The yielding stress of the epoxy resin determined from the tensile strength is 40.3 MPa. The openness of the crack in the epoxy substrate and nanocomposite is determined by using equation (6) as shown in Fig. 3.

The δ_c value of unmodified epoxy is about 6.03 μm . However, upon addition of nanosilica the openness of the crack in the nanocomposite gradually increase and reach a maximum of 16.38 μm at nanosilica content of 5%. Eventhough the presence of

nanosilica is one of reasons to increase the openness of the crack, but comparing with the nanosilica size of 20 - 40 nm this openness of the crack in the nanocomposite is significantly larger. Based on the rule of the pinning crack, the reinforcing particles are efficient when the particle size is larger than the openness of the crack. This indicates that the effect of the crack pinning of nano particles on the epoxy toughening is small in comparison with micron particles.

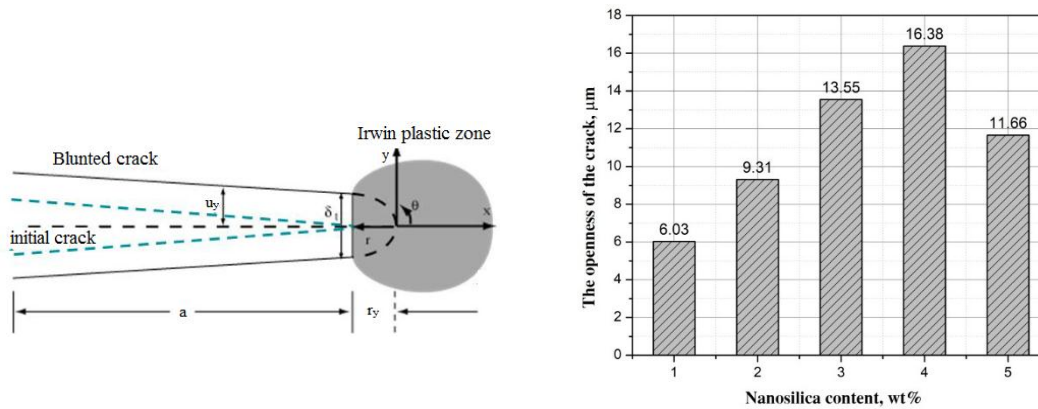


Fig. 3. Dependence of the openness of the crack on nanosilica contents.

3.2.3. Plastic deformation

Plastic deformation is the characteristic state of the materials, which varies the shape under impact of the stress and recovers to the initial shape when removal of the stress. Because of 3D network sewing property, thermoset plastic has capability of plastic deformation resistance much higher than thermoplastic. However, under the impact of the external force, they still can be deformed even plasticity. The slippery plasticity have been studied on the epoxy-based composite by several researchers [14], which was employed in this work to improve the toughening strength of epoxy-nanosilica nanocomposite. This mechanism includes the elongation deformation of the polymer substrate and the formation of ambient voids of nanoparticles under the impact of 3D stress near the crack peak. Kinloch [19] propose the calculating equation for the plastic deformation area in front of the crack peak under the planar deformation condition, it depends on the destructive toughness (K_{IC}) and the yielding stress (σ_c) of the polymer substrate.

$$r_y = \frac{1}{6\pi} \left[\frac{K_{IC}}{\sigma_c} \right]^2 \quad (7)$$

In the case of hypothesis that nanoparticles do not have chemical reaction with the substrate and do not change the polymer structure, the yielding stress is a constant. The

radius of the plasticity area is determined by equation (7), results are shown in Fig. 4. Figure 4a indicates that the pristine epoxy has $r_y = 30.75 \mu\text{m}$, upon addition of nanosilica the r_y linearly increase with nanosilica content in range of 1 - 5% and the r_y reaches a maximal value of $97.81 \mu\text{m}$ at 5%. When further content of silica is added, the plasticity area is declined ($r_y = 65.9 \mu\text{m}$). This decrease in the plasticity area upon high addition of silica is ascribed to the aggregation of nanoparticles into micron particles by Van der Waals force and hydrogen bonding. However, these bondings are facially broken under the external forces.

The spreading of the plastic deformation area in the presence of nanosilica will prevent the focus of the local stress and allow the material sustained a higher load before destruction. The dependence of r_y on $(K_{IC}/\sigma_y)^2$ with various nano contents is shown in Fig. 4b. The 'a' value of the regression equation is 0.0531, which is similar to Irwin model $(1/6\pi) 0.053$. This confirms that the spreading of the plastic deformation area at the crack tip is the main toughness mechanism in the nanocomposite.

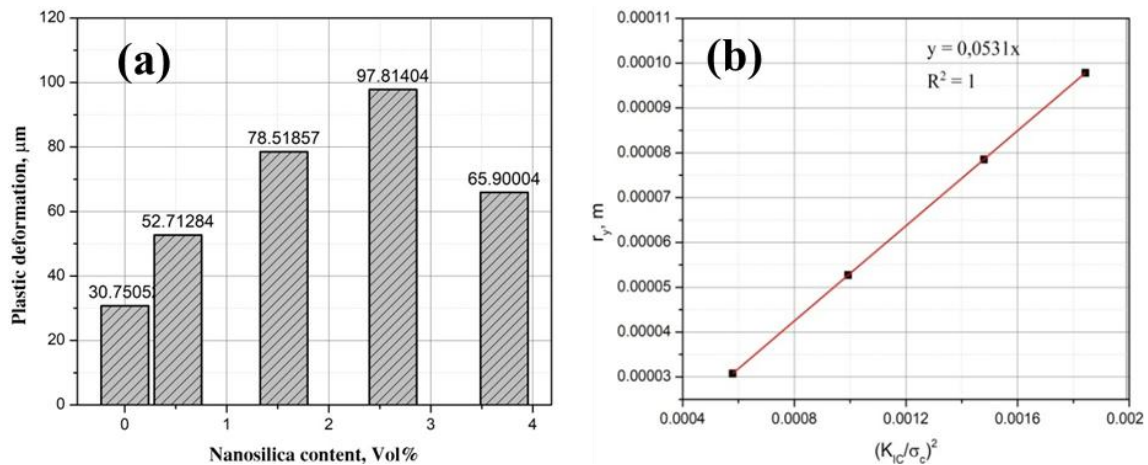


Fig. 4. Dependence of the plastic deformation area on nanosilica contents (a) and (b) $(K_{IC}/\sigma_y)^2$ ratio.

3.2.4. The spreading mechanism of voids and debond

This mechanism starts with voids, cracks or defects in the internal substrate phase and new voids generated during the bonding break down between nanoparticles and plastic substrate at the yielding stress, then external forces continue to impact around the voids to sketch, spread their sizes. This process reduces the stress in all three dimensions at surrounding areas, leads to the separation of neighboring particles more difficult.

FE-SEM image of the destructive surface after the destructive toughening test is displayed in Fig. 5. It is obvious from the figure that there is separation of nanoparticles out of polymer substrate and the spreading of voids surrounding nanosilica. The bright area in the FE-SEM image characterize for plastic deformation of the substrate phase. These evidences clearly confirm the appearance of the spreading mechanism of voids, which starts with the break bonding down of nanosilica against epoxy substrate. This process is characterized by statistic, which indicates that not all nanosilica particles were separated out of the substrate. The destruction energy of material involves in two processes: the first is the debonding between particles and substrat, and the second is the spreading of the voids. For the thermoset plastic, which strongly bond to nanoparticles, the energy needed to separate particles and spread the voids in the nanocomposite is remarkably high in comparison with the pristine substrate. These results are in good agreement with previous studies by Pearson, Mehrzad, and Mortezaei [6, 20, 22]. It is also obvious from the FE-SEM image that there is now characteristic bending area in the crack pinning mechanism.

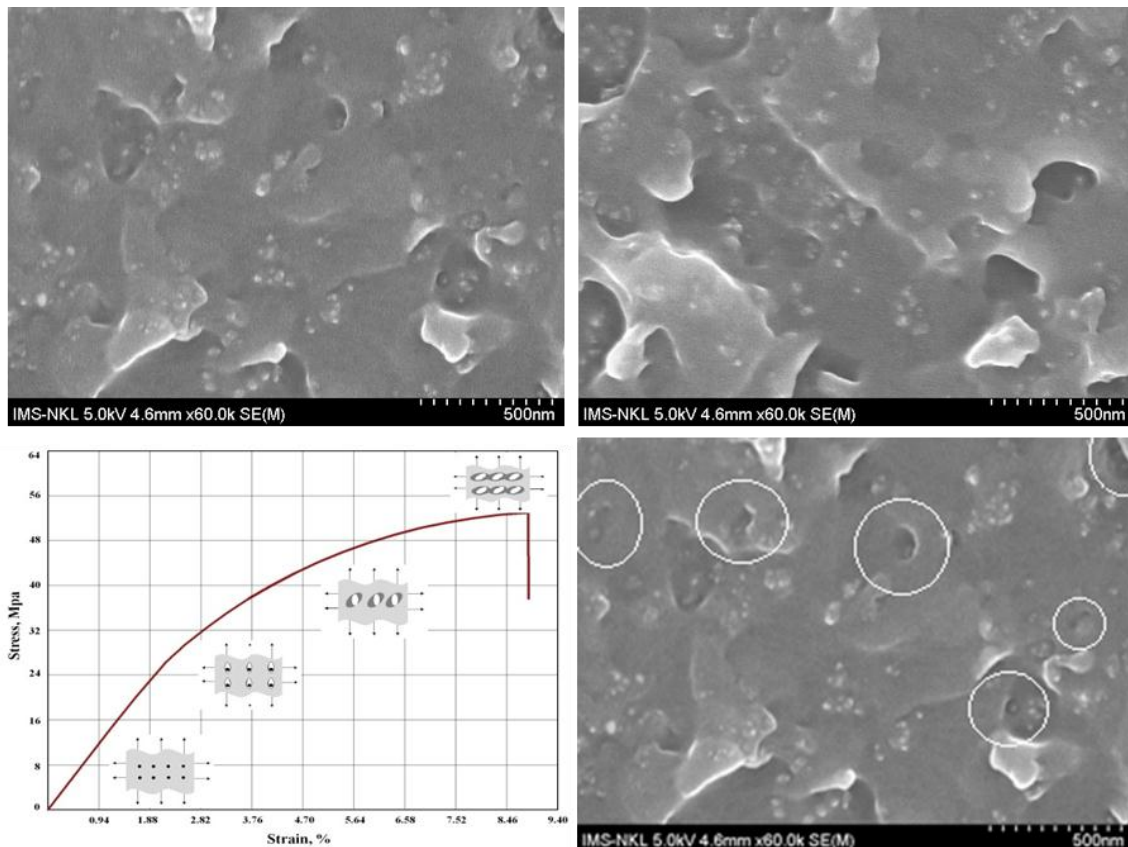


Fig. 5. Surface SEM image of destructed sample and the spreading model of voids under increase of stress in nanocomposite.

4. Conclusion

The use of nanosilica is to improve the mechanical and the creep strength for the epoxy. In the presence of nanosilica, nanocomposite has low deformation under external force and tends to recovery quicker than neat epoxy resin after removal of stress. Furthermore, the reasonable enhancement of destructive energy indicates that the nanocomposite changes from brittle to toughening state.

In this study, the toughening mechanism reveals that the crack deflection and pinning mechanism only contribute partially in the toughening of nanocomposite as the size of nanoparticles is too small compared to micron size of the crack tip. The surface SEM image of nanocomposite after destruction indicates the clear evidence of nano debonding out of substrate and the void spreading around nanoparticles. The plastic deformation in polymer was also clearly observed, which shows that the mechanism of plastic deformation, nanoparticles debonding and voids sketching are considered as the main factors to improve the mechanical strength and toughening in nanocomposite.

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ĐỘ BỀN RÃO VÀ CƠ CHẾ DẠI HÓA CỦA NANOCOMPOSITE EPOXY-SILICA

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Tóm tắt: Bài báo trình bày một số kết quả nghiên cứu hệ nanocomposite trên cơ sở nhựa epoxy/nanosilica biến tính với hợp chất cơ titan, đóng rắn với tetrabutyl titanate. Độ bền rão và khả năng hồi phục của hệ khi có m-nanosilica (1 - 7%) đã được nghiên cứu. Việc sử dụng nanosilica có tác dụng nâng cao độ bền cơ học và độ bền rão cho nhựa epoxy, khi có mặt nanosilica hệ nanocomposite có độ biến dạng thấp khi ngoại lực tác dụng và hồi phục nhanh hơn hệ nhựa epoxy ban đầu khi loại bỏ ứng suất, đồng thời sự tăng mạnh năng lượng phá hủy cho thấy nanocomposite có xu hướng chuyển trạng thái từ giòn sang dai. Các nghiên cứu cơ chế dai hóa cho thấy cơ chế chuyển hướng và ghim giữ vết nứt chỉ đóng góp một phần nhỏ trong việc dai hóa của nanocomposite, nguyên nhân do hạt nano quá nhỏ so với kích thước cỡ micron của đỉnh vết nứt. Ảnh kính hiển vi điện tử quét bề mặt nanocomposite sau khi phá hủy đã cho thấy bằng chứng rõ ràng của sự nhỏ đứt (debonding) các hạt nano ra khỏi nền và sự mở rộng lỗ trống xung quanh các hạt nano. Sự biến dạng dẻo trong polymer cũng được quan sát thấy rõ ràng, điều này cho thấy cơ chế biến dạng dẻo, tách hạt nano khỏi nền và kéo giãn các lỗ trống được nhận định là nguyên nhân chính làm tăng độ bền cơ học và khả năng dai hóa cho nanocomposite.

Từ khóa: Epoxy; nanosilica; KR-12; độ bền rão; cơ chế dai hóa.

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