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Mechanical properties of epoxy-based composites using nanoclays

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Abstract

Various percentages of nanoclay added into the epoxy resin were mixed by mechanical stirring and then mould forming to a dog-bone shape samples. The mechanical properties of the composites were characterized in terms of tensile tests and Vickers' hardness tests. Moreover, X-ray diffraction (XRD) and scanning electron microscopy (SEM) were employed to investigate the quantity and the distribution of nanoclay inside the composites. From the XRD patterns, the height of the diffraction peaks of nanoclay in the nanoclay–epoxy samples increased linearly with the weight percentage of nanoclay added. It was found that the composition at 5 wt% nanoclay gave the higher ultimate tensile strength, when compared with pure epoxy sample by an increment of about 5%. Furthermore, it gave the largest Vickers hardness value amongst all the compositions. However, the ductility decreased drastically and the sample was abruptly torn apart right after showing the peak of ultimate tensile strength. Furthermore, from the SEM images, the more the addition of nanoclay inside the epoxy, the smaller the fracture pieces in the cleavage surface. Therefore, it can be concluded that the ribbon-like nanoclay as shown in the SEM image hindered the linking up of epoxy chains network in decreasing the ductility, but it did make the nanoclay–epoxy samples stronger and harder in a certain extent.

Keywords: Nanoclays; Nanocomposites

1. Introduction

Montmorillonite is classified as magnesium aluminum silicate which has a sheet morphology, and can be used to make a new class of clay/polymer nanocomposites. The total surface area of montmorillonite can be as large as 750 m^2/g and the high aspect ratio (70–150) contributes to its enormous rheology benefits. In order to make the clay into organoclay, this can be achieved by reacting them with organ-cationic surfactants [1–3]. The modified clays then change from hydrophilic to organophilic. The organically modified clays are able to impart various rheological characteristics to organic polymer.

When a small amount of the organoclay like 4 wt% [4–7] is added into polymer, there is much increase in mechanical properties including tensile strength, Young's and flexural moduli. Therefore, composites can be made lighter and more transparent. Moreover, the heat resistant property of organoclay can increase the heat distortion temperature [8] of the composites by making them more dimensionally stable and flame retardant [9]. On the other hand, the high aspect ratio of the organoclay provides a tortuous path which makes difficulty for gas and vapor passing through. Therefore, the barrier properties can be greatly improved.

2. Experimental procedures

The epoxy resin Araldite®GY 251 and the hardener HY 956 from CIBA-GEIGY were used and mixed in the ratio of 4:1. The nanoclay was courtesy from Southern Clay Product Garamite 1958. Totally 10 compositions were prepared in order to show a gradual trend of changes upon mechanical and microstructural investigations, they were pure epoxy, that is 0 wt% nanoclay, 0.5 wt%, 1 wt%, 2 wt%, 3 wt%, 4 wt%, 5 wt%, 6 wt%, 7 wt% and 8 wt% nanoclay.

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The nanoclay was firstly introduced into the epoxy resin and mixed well with a glass rod before subjected to mechanical stirring at 500 rpm for 1 h. Secondly, the clay–resin mixture was poured out into a beaker and heated up in a hot water bath at 50 °C to lower down the viscosity. Then it was subjected into vacuum to get rid of air bubbles get trapped from mechanical stirring. After that the mixture was cooled down, the appropriate amount of hardener according to the above said mixing ratio was added and mixed well. Finally the whole mixture was poured inside a dog-bone shape mould for self curing and forming. The samples were taken out from the mold after 12 h and stayed for 1 week before subjected to various testings.

Tensile tests were performed in all 10 sets of samples and 5 samples from each set were tested for averaging. MTS machine Alliance RT/50 was employed for tensile test at a speed of 2 mm/min. For the microhardness test, microhardness tester FM-7E from Future Technology, Tokyo Japan was employed. Ten indentations from each set of samples were performed for averaging at 50 gf for 15 s. Since the finished surface of the sample was shiny plane after molding, the whole sample was subjected to XRD 2θ scanning from 5° to 80°, at 0.04° step size for 1 s per step. The fracture surface of the sample after tensile test was cut out and gold coated before subjected to SEM microstructural investigation.

Therefore, four different sets of testings were performed. The tensile test and the microhardness test were employed to investigate for the ultimate tensile strength and the microhardness value respectively, while XRD and SEM were used to check for the quantity and the microstructural distribution of nanoclay inside the nanoclay–epoxy samples respectively.

3. Experimental results and discussions

3.1. X-ray diffraction (XRD)

From the XRD patterns of nanoclay and the samples from 0 wt% to 8 wt% as shown in Fig. 1, it was found that as the nanoclay weight percentage in the nanoclay–epoxy samples increased, the height of the diffraction peaks of nanoclay in the nanoclay–epoxy samples also increased, but the peak positions remained the same as the pure nanoclay diffraction peaks. This indicated that this kind of nanoclay (Garamite 1958) acted like a nanodispersion over the whole epoxy matrix as there was no peak shifting.

The broad peak at 2θ about 20° in all the XRD patterns in Fig. 1 was due to the epoxy when comparing with the pure epoxy XRD pattern. The height of the diffraction peaks of nanoclay in the nanoclay–epoxy samples at 2θ about 7° increased linearly with the weight percentage of nanoclay from 0.5 wt% to 8 wt%, showing that there was no great loss of nanoclay during this fabrication process of the samples in any specific weight percentage. All the compositions can reflect their exact weight contents in a certain extent.

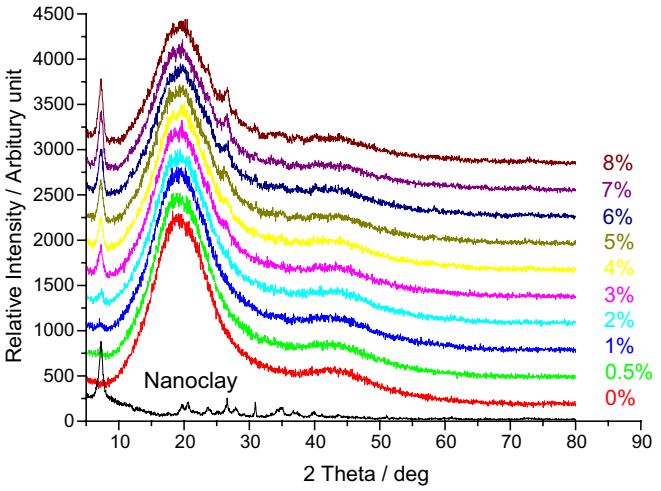

Fig. 1. XRD patterns of pure nanoclay and samples from 0 wt% to 8 wt%, scanned from 5° to 80° in 2-θ scan.

3.2. Tensile test

From the tensile test results as shown in Fig. 2, as the weight percentage of nanoclay increased, the ultimate tensile strengths of the samples also increased by a little. The 4 wt%, 5 wt% and 6 wt% samples showed the maximum increment by 5% in the ultimate tensile strength when compared with that of pure epoxy sample. However, the ductility dropped gradually starting from 1 wt% sample and dropped drastically by more than 70% for 8 wt% sample, when compared with that of pure epoxy sample.

A graph of the ultimate tensile strength of samples as a function of weight percentage of nanoclay in epoxy from 0 wt% to 8 wt% is shown in Fig. 3 for the ease of comparison. However, the reason for the sudden drop in ultimate tensile strength in 2 wt% was unknown. For samples well below 6 wt%, they could show the peak of a typical tensile curve, meaning they had gone through necking in a certain

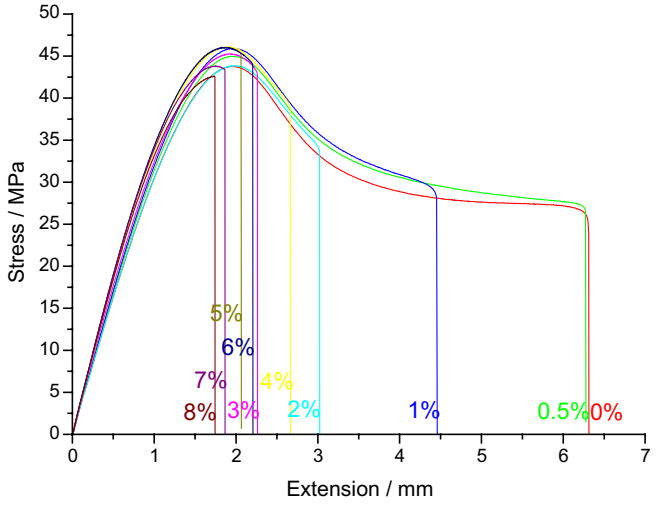

Fig. 2. The tensile curves of samples from 0 wt% to 8 wt% nanoclay, tested at a strain rate of 2 mm/min.

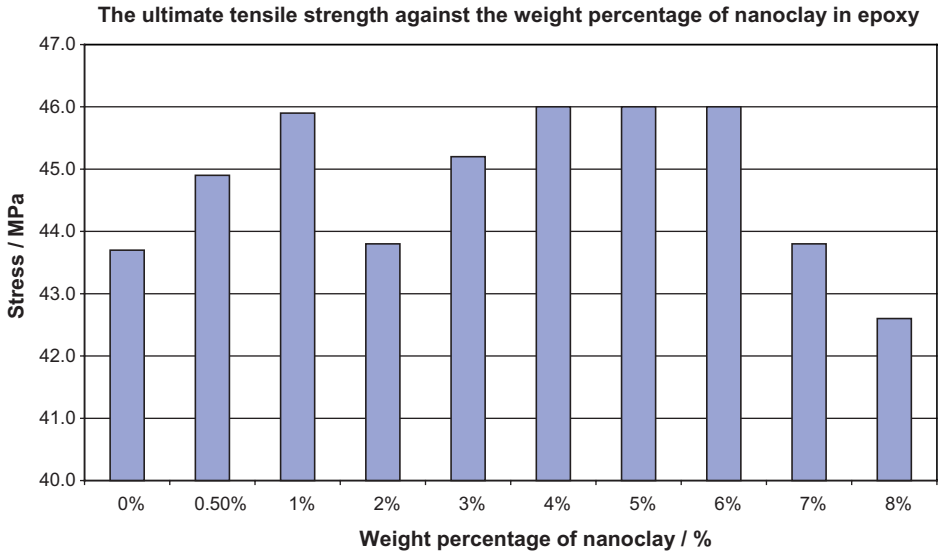

Fig. 3. The ultimate tensile strength of samples as a function of weight percentage of nanoclay in epoxy from 0 wt% to 8 wt%.

extent. But it was not the case for 7 wt% and 8 wt%, instead they tended to break apart before the peak, which meant they were brittle.

The drop in the ultimate tensile strength for 7 wt% and 8 wt% was mostly due to the big holes existed inside the nanoclay–epoxy samples. Those big holes were created when mixing the clay–resin mixture with hardener. It was because when the nanoclay weight percentage increased, the mixture itself became too viscous and sluggish. The more the nanoclay added, the more viscous of the clay–resin mixture. Once the hardener was added in and mixed together, big bubbles were easily created and trapped inside the sluggish mixture. When they were left inside the mold for self curing and forming, the finished products would become full of bubbles. Later on, when it was tested under MTS machine, the bubbles inside could not withstand the force and might initiate cracks to propagate throughout the sample, that was the reason why these two percentages showed smaller ultimate tensile strengths and the failure was catastrophic.

3.3. Vickers' hardness measurement

From the Vickers' hardness measurements, it showed similar results as the tensile tests, since there is a co-relationship between the hardness and the tensile strength in term of mechanical property. The 5 wt% sample showed the largest HV value of 11.3 when compared with that of pure epoxy sample of 9.8. A graph of the Vickers hardness value of samples as a function of weight percentage of nanoclay in epoxy from 0 wt% to 8 wt% is shown in Fig. 4.

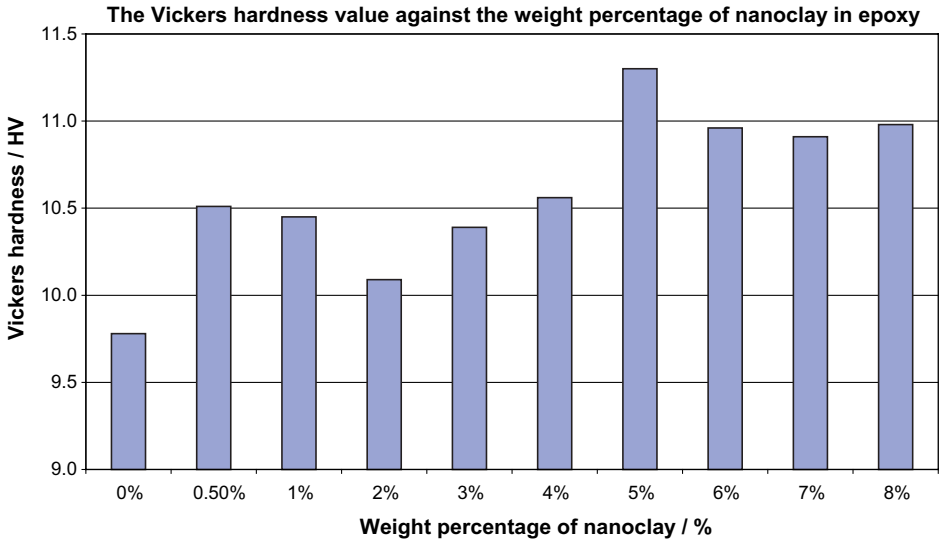

Fig. 4. The Vickers' hardness value of samples as a function of weight percentage of nanoclay in epoxy from 0 wt% to 8 wt%.

3.4. Scanning electron microscopy (SEM)

From the SEM image as shown in Fig. 5, the nanoclay was shown as nanoribbon with about several microns in length and about 200 nm in width. It was not hard to imagine when this kind of nanoribbon was evenly distributed across the matrix, it would inter-lock and entangle with the polymer chains in the matrix.

For the ease of comparison, except the SEM image of nanoclay, the SEM images of 0 wt%, 2 wt%, 4 wt%, 6 wt% and 8 wt% nanoclay–epoxy samples were all focused to a magnification of 10,000×. For the SEM image of pure epoxy sample as shown in Fig. 6, the cleavage surface was cleared and planed, thin lines or cracks were hardly seen. For the 2 wt% nanoclay–epoxy sample as shown in the SEM image in Fig. 7, the cleavage surface was a big differ-

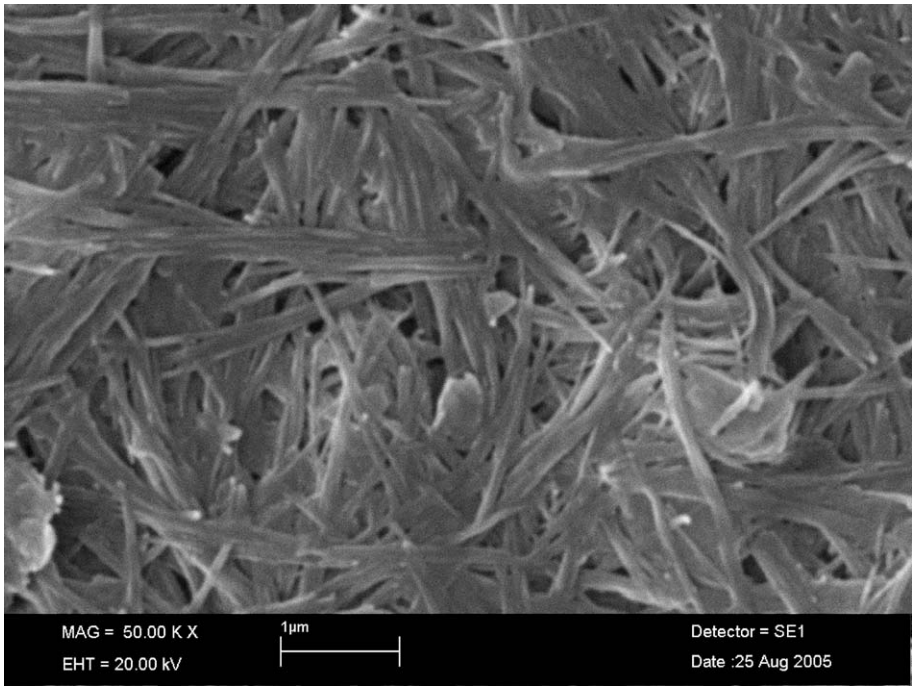

Fig. 5. The SEM image of nanoclay magnified in 50,000×.

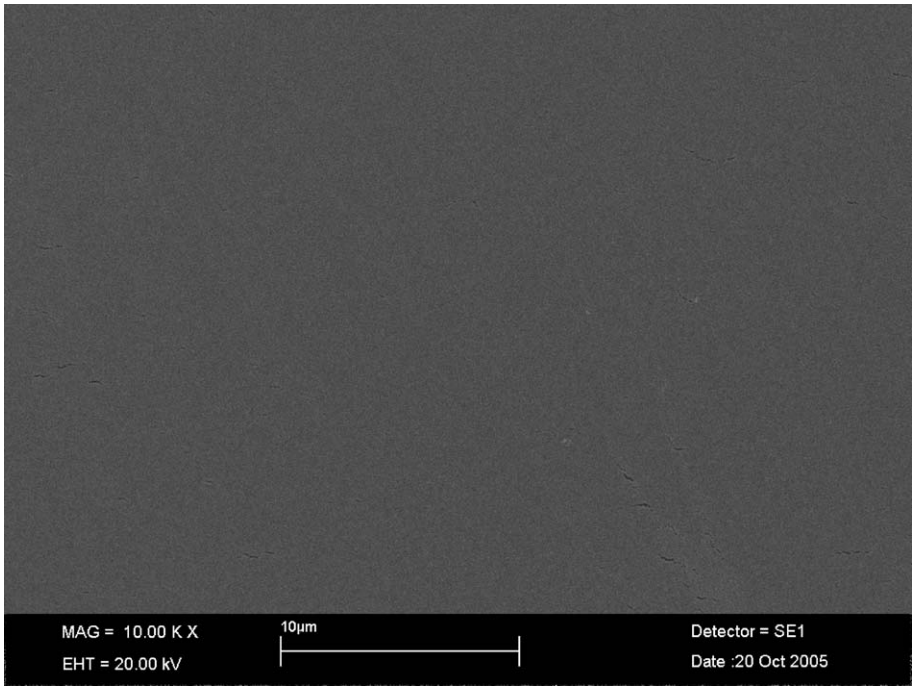

Fig. 6. The SEM image of pure epoxy (0 wt% nanoclay) magnified in 10,000×.

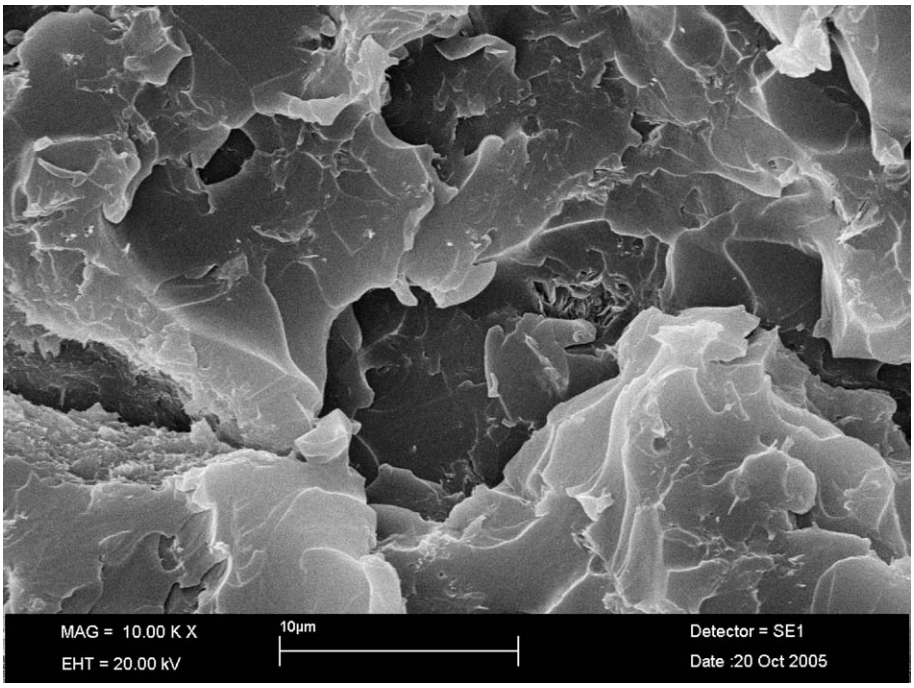

Fig. 7. The SEM image of 2 wt% nanoclay magnified in 10,000×.

ent from that of pure epoxy sample. The cleavage surface showed fracture pieces with smooth white facture borders.

In general, for the SEM images of 4 wt%, 6 wt% and 8 wt% nanoclay–epoxy samples as shown in Figs. 8–10 respectively, there was a trend that the cleavage surface broken with smaller and rougher fracture pieces. Sometimes, some tiny white lines, which believed to be the nanoclay (the nanoribbon), were found coming out from the fracture surfaces.

Therefore, the nanoclay introduced inside the epoxy acted like the grid lines of a net, When the weight percentage of nanoclay inside the epoxy samples increased, as if the number of grid lines of the net increased, the sieve holes of the net became smaller and smaller. That is why the frac-

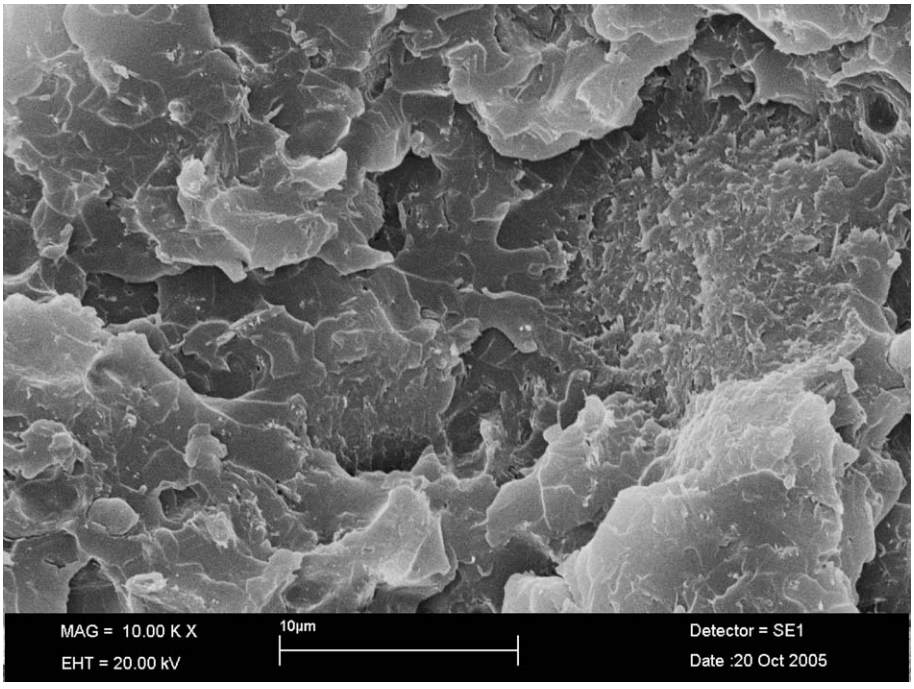

Fig. 8. The SEM image of 4 wt% nanoclay magnified in 10,000×.

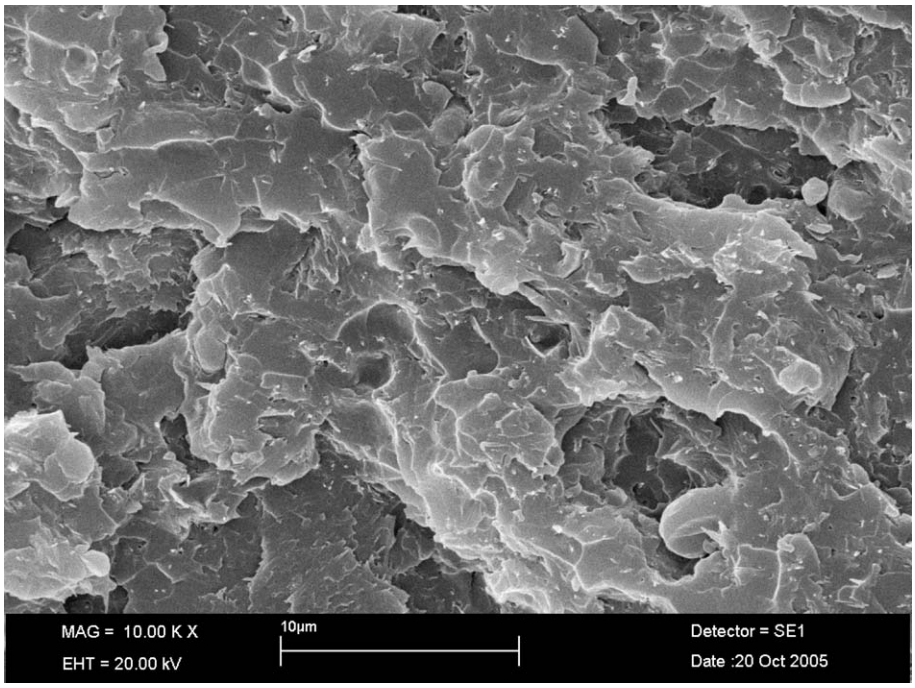

Fig. 9. The SEM image of 6 wt% nanoclay magnified in 10,000×.

ture pieces became rougher and smaller in samples with higher nanoclay contents.

When generalizing the pictures of SEM images with tensile test results, the addition of this kind of ''nanoribbon'' into the epoxy matrix, the nanoribbon would interlock with the epoxy network chains. With the addition of higher nanoclay content, the interlocking mechanism seemed prevail more comprehensively. Therefore, it seemed that the introduction of this kind of nanoclay would make the sample brittle, or on the other way of saying, the ductility decreased with the addition of this kind of nanoclay. The more the addition of nanoclay inside the epoxy, the more the brittleness of the samples. Therefore, it can be concluded that the ribbon-like nanoclay hindered the linking up of epoxy chains network, making the nanoclay–epoxy samples more brittle, but it

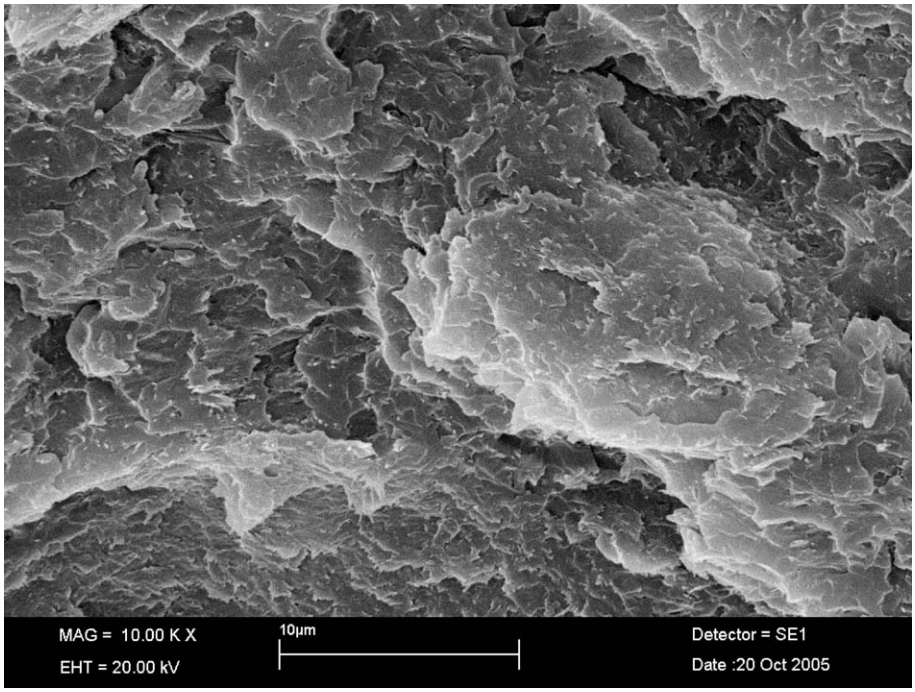

Fig. 10. The SEM image of 8 wt% nanoclay magnified in 10,000×.

did make the nanoclay–epoxy samples stronger and harder in a certain extent.

4. Conclusions

The addition of nanoclay into the epoxy matrix by mechanical stirring method was studied. Various mechanical tests and microstructural investigations were carried out. It was found that the 5 wt% nanoclay epoxy sample gave the highest ultimate tensile strength and Vickers' hardness value amongst all the nanoclay compositions from 0 wt% to 8 wt%. However, the ductility decreased drastically with the addition of higher nanoclay content inside the epoxy sample, which would cause catastrophic failure upon tensile testing [10]. Accounting for the ease of molding method in this paper applied, the nanoclay content should be kept below 5 wt% in order to get good mechanical properties [11].

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