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A comparative study to predict the interphase modulus in polymer nanocomposites

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ABSTRACT: In this study, the interphase modulus (E_i) in polymer nanocomposites is calculated by two methods and the calculated results are compared at different conditions. In the first method, the experimental moduli of samples are applied to Ji model and suitable "E_i" is calculated. In the second method, a multilayered interphase is considered, in which the Young's moduli of layers (E_k) depend to the distance between the nanoparticle surface and the polymer matrix by power function of "Y" parameter. The "E_i" is calculated for multilayered interphase assuming the same and different layer thicknesses (t_k) by Parallel and Series models. Finally, the "E_i" values calculated by the explained methods are compared for two reported samples. © 2016 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 44076.

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INTRODUCTION

The introduction of nanotechnology suggested that the nanoparticles incorporated in polymer matrixes can introduce a revolution in polymer science and technology. Nanoparticles have been extensively used in polymer matrix as reinforcement, due to several favorable properties that they induce compared to micro-fillers such as the high levels of stiffness and surface area at very low content.[1–6] In the recent years, polymer nanocomposites as cheap, nontoxic, easy-produced, high thermal resistant and strong materials show many applications in different fields. However, the main challenges in production of polymer nanocomposites include the effective techniques to control the dispersion of the nanoparticles in polymer matrix and promote the compatibility between the polymer and the nanoparticles.[7–10] Many studies in the literature have focused on material and processing parameters to improve the nanoparticles dispersion and enhance the interfacial interaction in polymer nanocomposites containing inorganic nanoparticles.

From a modeling view, the predictive methods provide much information for design and optimization of polymer nanocomposites.[11–13] The conventional models for microcomposites such as Guth, Mori-Tanaka and Halpin-Tsai were used to predict the Young's modulus of nanocomposites.[14,15] Nevertheless, they cannot suggest accurate predictions for nanocomposites, because they only consider the effects of constituent properties such as volume fraction and modulus on the final modulus, while the modulus of nanocomposites significantly depends to the nanoparticles size and interphase properties between the matrix and the nanoparticles.[16,17] The actual properties of nanocomposites should be assumed in modeling methods to understand the main relations between nanostructure and nanocomposite behavior.

The interphase properties cannot be directly measured from experiments, due to manipulation of the interphase size and the interfacial interactions at nanoscale. Accordingly, the theoretical approaches are much useful and helpful to quantify the properties of interphase. The interphase properties such as thickness, modulus, and strength can be determined by micromechanical models for mechanical properties such as Young's modulus and tensile strength.[18,19] Ji model[20] was effectively used to characterize the Young's modulus and thickness of interphase in polymer nanocomposites.[21–23] Also, the "B" parameter in the Pukanszky model for yield strength of nanocomposites can estimate the strength and thickness of interphase.[24] Many researchers also assumed a multilayered interphase, in which the properties of each layer are different from others.[25–28] Moreover, the influences of the various layer properties such as thickness and modulus on the nanocomposite behavior were discussed. For example, Shabana[25] studied the roles of the interphase thickness, number of layers, properties of each layer, progressive debonding damage, elasto-plasticity of the matrix and the size of reinforcement on the thermomechanical properties of nanocomposites. However, there is not any report, which compares the calculations of interphase properties assuming the

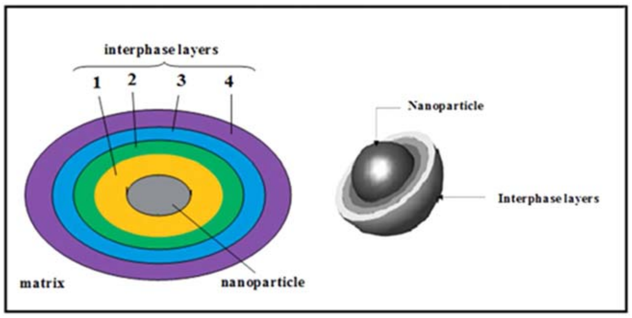

Figure 1. The illustration of interphase layers around the nanoparticles in polymer nanocomposites.[25,29] [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

micromechanical model and the multi-layered interphase. This comparison will produce useful results for interphase properties, which govern the final behavior of nanocomposites.

In this work, the interphase modulus (E_i) in polymer nanocomposites is calculated by two methods and the results are compared at different states. The experimental modulus is applied to Ji model and a suitable "E_i" is calculated. In addition, a multilayered interphase is considered, in which the Young's modulus of each layer (E_k) is changed from nanoparticle surface to polymer matrix. The "E_i" for multilayered interphase is calculated by Parallel and Series models at different "Y" parameter. Finally, the calculations of "E_i" are compared and some attractive results are presented.

FORMULATIONS

Interphase Layers

A multilayered interphase including n layers can be considered around the nanoparticles, in which the thermomechanical properties of layers are changed from nanoparticle surface to polymer matrix. The interphase layers are numbered from $x=0$ at nanoparticles surface to $x=t$ (interphase thickness) at polymer matrix (Figure 1).

The "x" for the central point of the kth layer (x_k) is given as:

$$x_k = kt_k - \frac{t_k}{2} \tag{1}$$

where "t_k" is the thickness of the kth layer. In our previous article,[29] it was reported that the Young's modulus of interphase layers changes by a power function as:

$$E_k = E_p - (E_p - E_m)\left(\frac{x_k}{t}\right)^Y \tag{2}$$

where "E_m" and "E_p" are the Young's moduli of polymer matrix and nanoparticles, respectively. In addition, "Y" is an interphase parameter, which shows the properties of interphase.

Same Thickness for All Interphase Layers

When the interphase layers have a same thickness, "t_k" is given by:

$$t_k = \frac{t}{n} \tag{3}$$

At this condition, the volume fraction of each layer (φ_k) is expressed[25] by:

$$\frac{\varphi_k}{\varphi_i} = \frac{(d+\sum_{j=1}^{k} 2t_j)^2(\alpha d+\sum_{j=1}^{k} 2t_j)-(d+\sum_{j=1}^{k-1} 2t_j)^2(\alpha d+\sum_{j=1}^{k-1} 2t_j)}{(d+2t)^2(\alpha d+2t)-\alpha d^3} \tag{4}$$

where "φ_i" is the total volume fraction of interphase in nanocomposite sample, "d" is the diameter or thickness of nanoparticles, and "α" is the aspect ratio of nanofiller. Assuming the spherical nanoparticles and a 5-layered interphase, the "φ_k" is calculated by:

$$\frac{\varphi_1}{\varphi_i} = \frac{(d+2t_1)^3-d^3}{(d+2t)^3-d^3} \tag{5}$$

$$\frac{\varphi_2}{\varphi_i} = \frac{(d+2t_1+2t_2)^3-(d+2t_1)^3}{(d+2t)^3-d^3} \tag{6}$$

$$\frac{\varphi_3}{\varphi_i} = \frac{(d+2t_1+2t_2+2t_3)^3-(d+2t_1+2t_2)^3}{(d+2t)^3-d^3} \tag{7}$$

$$\frac{\varphi_4}{\varphi_i} = \frac{(d+2t_1+2t_2+2t_3+2t_4)^3-(d+2t_1+2t_2+2t_3)^3}{(d+2t)^3-d^3} \tag{8}$$

$$\frac{\varphi_5}{\varphi_i} = \frac{(d+2t_1+2t_2+2t_3+2t_4+2t_5)^3-(d+2t_1+2t_2+2t_3+2t_4)^3}{(d+2t)^3-d^3} \tag{9}$$

Same "φ_k" for All Interphase Layers

The interphase layers can be assumed to have a same "φ_k" and different "t." In this condition, "φ_k" is calculated for each layer[25] by:

$$\frac{\varphi_k}{n\varphi_f} = \frac{(d+\sum_{j=1}^{k} 2t_j)^2(\alpha d+\sum_{j=1}^{k} 2t_j)-(d+\sum_{j=1}^{k-1} 2t_j)^2(\alpha d+\sum_{j=1}^{k-1} 2t_j)}{\alpha d^3} \tag{10}$$

where "φ_f" is the volume fraction of nanofiller in polymer nanocomposite. For spherical nanoparticles and a 5-layered interphase, "φ_k" is calculated in this state by:

$$\frac{\varphi_1}{5\varphi_f} = \frac{(d+2t_1)^3-d^3}{d^3} \tag{11}$$

$$\frac{\varphi_2}{5\varphi_f} = \frac{(d+2t_1+2t_2)^3-(d+2t_1)^3}{d^3} \tag{12}$$

$$\frac{\varphi_3}{5\varphi_f} = \frac{(d+2t_1+2t_2+2t_3)^3-(d+2t_1+2t_2)^3}{d^3} \tag{13}$$

$$\frac{\varphi_4}{5\varphi_f} = \frac{(d+2t_1+2t_2+2t_3+2t_4)^3-(d+2t_1+2t_2+2t_3)^3}{d^3} \tag{14}$$

$$\frac{\varphi_5}{5\varphi_f} = \frac{(d+2t_1+2t_2+2t_3+2t_4+2t_5)^3-(d+2t_1+2t_2+2t_3+2t_4)^3}{d^3} \tag{15}$$

In addition, the thickness of each layer (t_k) is expressed by:

$$t_k=0.5\left[\sqrt[3]{\frac{d^3\varphi_i}{n\varphi_f}+\left(d+\sum_{j=1}^{k-1}2t_j\right)^3}-d-\sum_{j=1}^{k-1}2t_j\right] \quad (16)$$

Assuming spherical nanoparticles and a 5-layered interphase, "t_k" is given by:

$$t_1=\left[\sqrt[3]{\left(\frac{\varphi_i}{5\varphi_f}\right)+1}-1\right]\frac{d}{2} \quad (17)$$

$$t_2=0.5\left[\sqrt[3]{\left(\frac{d^3\varphi_i}{5\varphi_f}\right)+(d+2t_1)^3}-d-2t_1\right] \quad (18)$$

$$t_3=0.5\left[\sqrt[3]{\left(\frac{d^3\varphi_i}{5\varphi_f}\right)+(d+2t_1+2t_2)^3}-d-2t_1-2t_2\right] \quad (19)$$

$$t_4=0.5\left[\sqrt[3]{\left(\frac{d^3\varphi_i}{5\varphi_f}\right)+(d+2t_1+2t_2+2t_3)^3}-d-2t_1-2t_2-2t_3\right] \quad (20)$$

$$t_5=0.5\left[\sqrt[3]{\left(\frac{d^3\varphi_i}{5\varphi_f}\right)+(d+2t_1+2t_2+2t_3+2t_4)^3}-d-2t_1-2t_2-2t_3-2t_4\right] \quad (21)$$

Models for Young's Modulus

In the previous section, the formulations for "φ_k" and "t_k" of each interphase layer were expressed for two states of constant "t_k" and "φ_k" in polymer nanocomposites. In addition, it was indicated that the modulus of each layer (E_k) can be obtained by eq. (2). Now, some models are suggested to calculate the overall modulus of interphase (E_i) by "E_k" and the experimental relative modulus of nanocomposites (E_R).

Assuming a uniform strain in interphase layers, "E_i" can be expressed by Rule of mixtures or Parallel model[14] as:

$$E_i=E_1\varphi_1 + E_2\varphi_2 + E_3\varphi_3 + E_4\varphi_4 + E_5\varphi_5 \quad (22)$$

In addition, assuming a same stress in interphase layers, the Series, or Inverse rule of mixtures model[14] for "E_i" is presented as:

$$\frac{1}{E_i}=\frac{\varphi_1}{E_1}+\frac{\varphi_2}{E_2}+\frac{\varphi_3}{E_3}+\frac{\varphi_4}{E_4}+\frac{\varphi_5}{E_5} \quad (23)$$

Ji *et al.*[20] also suggested a three-phase model for Young's modulus of nanocomposites, which gives the thickness and modulus of interphase by the properties of matrix, nanofiller, and interphase between polymer and nanoparticles. The Ji model for nanocomposites reinforced with the spherical nanoparticles is expressed as:

$$E_R=\left[(1-\alpha)+\frac{\alpha-\beta}{(1-\alpha)+\frac{\alpha(m-1)}{\ln(m)}}+\frac{\beta}{(1-\alpha)+\frac{(\alpha-\beta)(m+1)}{2}+\beta\frac{E_p}{E_m}}\right]^{-1} \quad (24)$$

$$\alpha=\sqrt{\left(\frac{2t}{d}+1\right)^3\varphi_f} \quad (25)$$

$$\beta=\sqrt{\varphi_f} \quad (26)$$

$$m=\frac{E_i}{E_m} \quad (27)$$

where "E_R" is relative modulus as E_c/E_m, "E_c" is the Young's modulus of nanocomposite. When the interphase is neglected in this model ($t=0$), the Ji model reduces to Takayanagi model as:

$$E_R=\left[(1-\alpha)+\frac{\beta}{(1-\alpha)+\beta\frac{E_p}{E_m}}\right]^{-1} \quad (28)$$

RESULTS AND DISCUSSION

In this part, the suggested models are applied to calculate the properties of interphase in two nanocomposite samples from literature. Afterwards, the final predictions of Parallel, Series, and Ji models for "E_i" are differed and the results are reported.

The methyl methacrylate copolymerized with 2-(methacryloyloxy) ethyltrimethylammonium chloride, P(MMA-*co*-MTC)/SiO_2 sample was selected from[30] in which 0.5, 1, 1.5, and 2 wt % of nanofiller was added. In this sample, "E_R" was reported as 1, 1.14, 1.35, 1.39, and 1.5. Moreover, $E_m=1.87$ and $E_p=80$ GPa are considered for this sample. A 5-layered interphase is taken into account for this sample at all nanofiller concentrations. "t" can be varied from 0 to 40 nm as the common range of gyration radius of macromolecules. However, an average value of 20 nm is assumed for "t." When a same thickness is considered for each interphase layer, "t_k" is calculated as 4 nm for a 5-layered interphase and the layer volume fractions (φ_k) are calculated by eqs. (5)–(9) and reported in Table I.

The "φ_k"for the layers near the nanoparticles is lower than that of near the matrix. The general modulus of interphase (E_i) can be obtained by Parallel [eq. (22)] and Series [eq. (23)] models by the values of "E_k" calculated by eq. (2) at different "Y" parameter. However, assuming $t=20$ nm in this sample, the "E_i" is obtained by applying the experimental Young's modulus to Ji model [eqs. (24)–(27). The values of "m" [eq. (27)] fitted to Ji model are calculated as 0, 7, 15, 8, and 8 for different "φ_f", which give the average "E_i" as 0, 13.09, 28.05, 14.96, and 14.96 GPa. Figure 2 demonstrates the average "E_i" calculated by Ji model and eqs. (22) and (23), when a same thickness is considered for each interphase layer. Figure 2(a) shows the calculations of the eqs. (22) and (23) at $Y=0.14$, while infinite "Y" ($Y\rightarrow\infty$) is considered in Figure 2(b).

In Figure 2(a), the predictions of eq. (23) can only fit to Ji model at 2 wt % of SiO_2. However, the predictions of Parallel model [eq. (22)] are below the Ji calculations. In addition, the models show dissimilar trends for "E_i" as the nanofiller content increases. Therefore, the suggested models calculate different data for "E_i" in this sample at same "t_k". The predictions of the Parallel and Series models at a maximum value of "Y" ($Y\rightarrow\infty$) are also estimated. It is observed that the Series model causes

Table I. The Characteristics of the Interphase Layers in P(MMA-*co*-MTC)/SiO_2 Nanocomposite

Layers properties	0.5 wt % (0.0027 vol %)	1 wt % (0.0054 vol %)	1.5 wt % (0.0081 vol %)	2 wt % (0.0108 vol %)
φ_1	0.0047	0.0094	0.0141	0.0189
φ_2	0.0083	0.0166	0.0250	0.0334
φ_3	0.0129	0.0260	0.039	0.0521
φ_4	0.0186	0.0373	0.0561	0.0750
φ_5	0.0253	0.0508	0.0764	0.1020
t_1	8.3790	8.3671	8.1076	7.7769
t_2	4.1380	4.1340	4.0472	3.9353
t_3	3.0052	3.0025	2.9443	2.8693
t_4	2.4271	2.4250	2.3797	2.3213
t_5	2.0660	2.0642	2.0264	1.9778

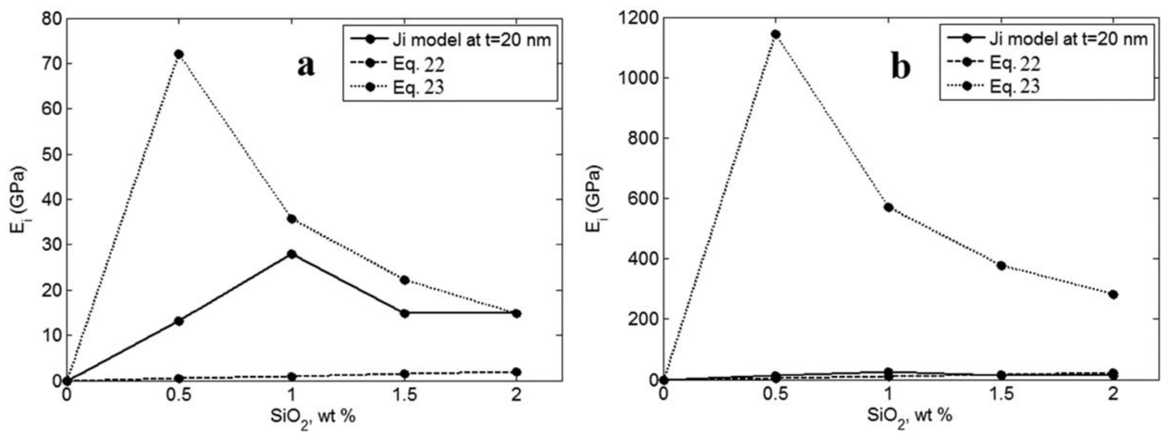

Figure 2. "E_i" calculated by Ji model and eqs. (22) and (23) for P(MMA-*co*-MTC)/SiO_2 sample assuming a same thickness for interphase layers: (a) $Y = 0.14$ and (b) $Y \to \infty$.

very high calculations for "E_i". In addition, the predictions of Parallel model cannot well fit to the calculations of Ji model. As a result, the calculated moduli by the suggested models at a high range of "Y" cannot show similar values assuming a similar "t_k".

The calculations of the suggested models are also examined when a same "φ_k" is considered for all interphase layers. Assuming an average value of "t" as 20 nm, the total "φ_i" is roughly calculated as 0, 0.07, 0.14, 0.2, and 0.25 vol % at different nanofiller contents (indicated above). The "φ_k" for each

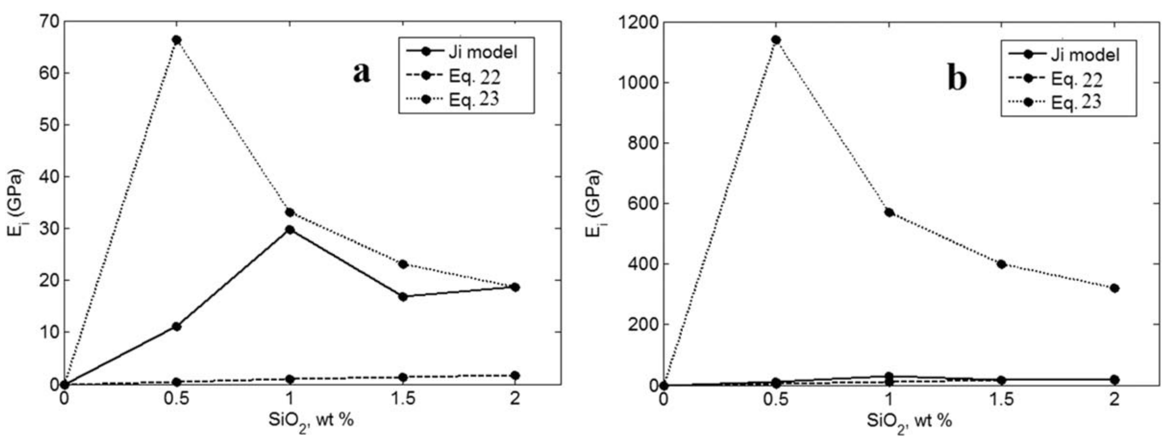

Figure 3. The predicted "E_i" by the suggested models for P(MMA-*co*-MTC)/SiO_2 sample assuming different "t_k": (a) $Y = 0.125$ and (b) $Y \to \infty$.

Table II. The Characteristics of the Interphase Layers in PP/$CaCO_3$ Nanocomposite

Layers properties	2 wt % (0.0068 vol %)	4 wt % (0.0137 vol %)	6 wt % (0.02 vol %)
φ_1	0.0056	0.0118	0.0153
φ_2	0.0081	0.0171	0.0222
φ_3	0.011	0.0234	0.0303
φ_4	0.0144	0.0307	0.0397
φ_5	0.0183	0.0389	0.0503
φ_6	0.0226	0.048	0.0623
t_1	9.6352	9.9686	8.8597
t_2	5.85	5.9906	5.5152
t_3	4.4383	4.5322	4.2142
t_4	3.6568	3.7291	3.4841
t_5	3.1487	3.2085	3.0062
t_6	2.7872	2.8386	2.6647

layer is calculated as $\varphi_i/5$. In addition, the thickness of each layer is calculated by eqs. (17)–(21) and shown in Table I. It is found that the thickness of interphase layer decreases as the number of layer increases. The "E_k" for each layer can be calculated by the thickness of each layer and "Y" value [eq. (2)] and the overall "E_i" is obtained by eqs. (22) and (23). Additionally, the Ji model gives the average "E_i" at each "φ_f" by fitting the experimental moduli to Ji model. The "m" values are calculated as 0, 6, 16, 9, and 10 in different "φ_f", which cause "E_i" as 0, 11.22, 29.92, 16.83, and 18.7 GPa.

Figure 3 shows the calculations of the proposed models at different "Y" parameter. The calculations of Ji model is only fitted to the Series model at the very high content of SiO_2 and $Y = 0.125$, but the predictions of Ji model are between the calculations of Parallel and Series models at other "φ_f". Moreover, to evaluate the effect of very high "Y" value on the predictions of "E_i", the results of the Parallel and Series models at $Y \to \infty$ together with the predictions of Ji model are shown in Figure 3(b). It is observed that the Series model significantly overpredicts the "E_i" at this condition. However, the Parallel model can cause similar predictions to Ji model, but it cannot carefully predict the trend of the Ji calculations for the above sample. Accordingly, the calculations of "E_i" by the suggested methods do not show any similar data in polymer nanocomposites. It should be noted that considering a same or different "t_k" for interphase layers do not considerably change the predictions of the Ji, Parallel, and Series models at similar "Y" values (see Figures 2 and 3).

To confirm the above evidences and evaluate the predictions of above models, another sample at different conditions is studied. The polypropylene (PP)/$CaCO_3$ sample containing 2, 4, and 6 wt % of $CaCO_3$ was chosen from the work of Chen *et al.*[31] In this sample, the "E_R" was measured as 1, 1.07, 1.1, and 1.27 at indicated "φ_f", "E_p" is about 26 GPa and $E_m = 1.75$ GPa. An interphase thickness of 30 nm as well as a 6-layered interphase is considered at all nanofiller contents. At the same "t_k" for all interphase layers, $t_k = 5$ nm is obtained for each layer. Similar to the explained trend for P(MMA-*co*-MTC)/SiO_2 sample, the "φ_k" values are calculated and presented in Table II. Furthermore, the modulus of each layer [eq. (2)] and the general modulus of interphase by Parallel [eq. (22)] and Series [eq. (23)] models are obtained. However, assuming $t = 30$ nm in this

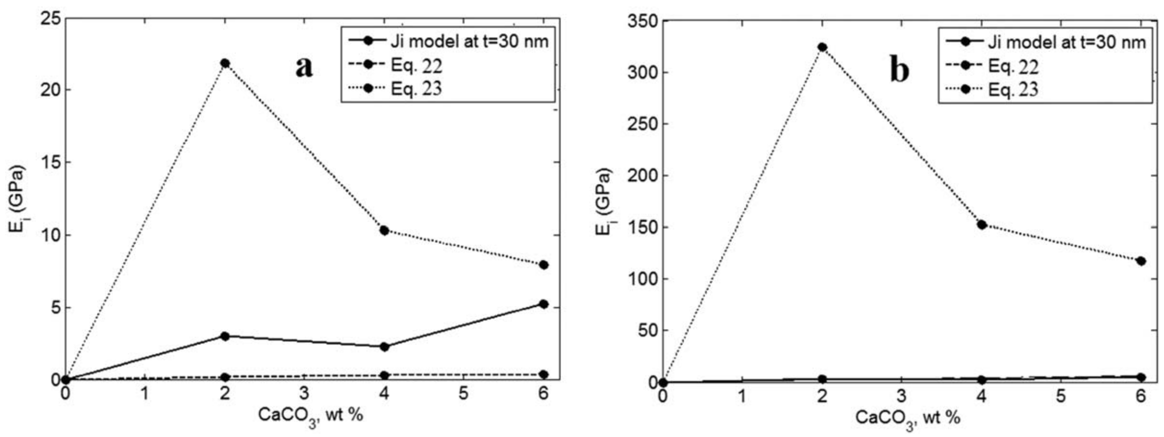

Figure 4. "E_i" calculations by Ji, Parallel, and Series models for PP/$CaCO_3$ sample assuming same thickness for interphase layers: (a) $Y \to 0$ and (b) $Y \to \infty$.

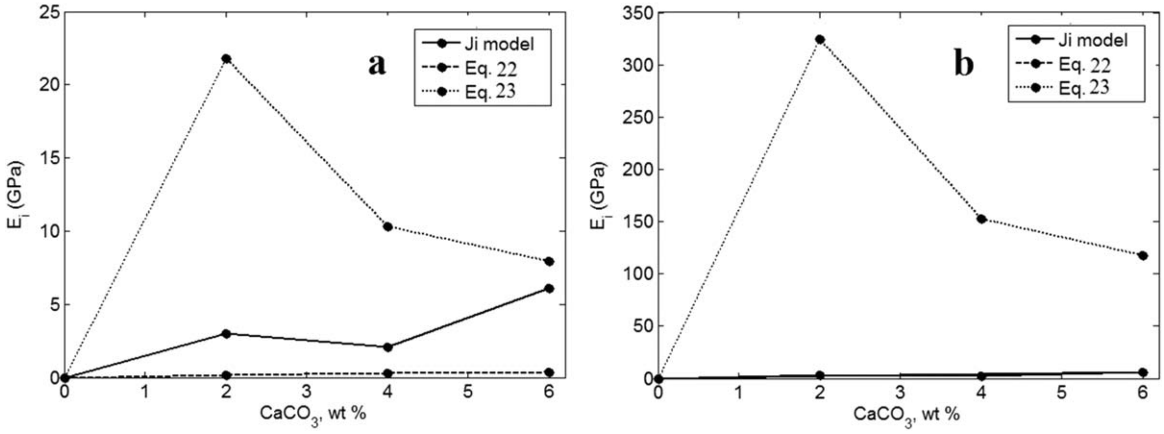

Figure 5. The predictions of "E_i" by different models for PP/$CaCO_3$ sample assuming different "t_k": (a) $Y\rightarrow 0$ and (b) $Y\rightarrow\infty$.

sample, the average "E_i" can be calculated by applying the experimental Young's modulus to Ji model.

Figure 4 shows the predictions of Series, Parallel, and Ji models at very low ($Y\rightarrow 0$) and very high ($Y\rightarrow\infty$) values of "Y". It is observed that the calculations of Ji model are between the predictions of Parallel and Series models at $Y\rightarrow 0$. The Parallel model can present the similar results to Ji model at very high "Y," but the differences between Ji and parallel models are not acceptable. As a result, the proposed models cannot show similar values for "E_i" at different "Y" levels from 0 to ∞, when a same thickness is considered for interphase layers. It is also understood that the most predictions are calculated by Series model at different levels of "Y" parameter. This trend is not similar to polymer nanocomposites, where the Parallel and Series models overpredict and underpredict the Young's modulus of polymer nanocomposites, respectively.[14] The most values of "E_i" by Series model may be attributed to the very low levels of "φ_k" in polymer nanocomposites (see Tables I and II).

The calculations of the proposed models are also evaluated when a same "φ_k" is considered for the interphase layers in PP/$CaCO_3$ sample. In this condition, different values for "t_k" are obtained. Assuming the total "φ_k" as 0, 0.08, 0.17, and 0.22 vol %, the "t_k" for all "φ_f" are calculated and shown in Table II. Furthermore, the "E_k" and "E_i" are calculated by the above explanation. In addition, the experimental data are fitted to Ji model and "E_i" is calculated.

Figure 5 depicts the calculations of the suggested models at this condition at different levels of "Y". Similar to Figure 4, the calculations of Ji model are between the calculations of eqs. (22) and (23) at $Y\rightarrow 0$. Also, very high levels of "E_i" is obtained by Series model at $Y\rightarrow\infty$. Accordingly, the calculations of the mentioned models cannot show a consistency even by assuming the different thicknesses for interphase layers.

The proposed models (Ji, Parallel, and Series) show different "E_i" values in different conditions (same and various thicknesses of interphase layers), while all models consider the properties of matrix, nanofiller, and interphase in their predictions. This occurrence may be related to this fact that the Ji model calculates the "E_i" by the experimental results of "E_R" [see eq. (24)], while the predictions of Parallel and Series models depend to "x_k" [see eq. (2)].

CONCLUSIONS

The interphase modulus (E_i) in polymer nanocomposites was calculated by fitting the experimental modulus to Ji model and assuming a multilayered interphase. In the first method, the experimental data were applied to Ji model and "E_i" was calculated at constant "t." In the second method, the Young's modulus of each layer (E_k), changed from nanoparticle surface to polymer matrix was calculated at different "Y" values and "E_i" was calculated by Parallel and Series models. The effects of the same and different "t_k" on the final "E_i" were also investigated.

The suggested models were applied to calculate the "E_i" in two examples from valid literature. The results indicated that the "E_i" calculated by all models show different levels, due to the different dependencies of "E_i" to effective parameters. The predictions of Ji model were between the calculations of Parallel and Series models at low "Y," but the Ji calculations were near the calculations of Parallel model at very high "Y." It was also found that the most predictions are calculated by Series model at different levels of "Y" parameter, while this model underpredicts the final Young's modulus of the polymer nanocomposites. This dissimilarity was attributed to the very low concentration of interphase layers (φ_k) in polymer nanocomposites.

Since the "E_i" levels by Parallel and Series models depend to different parameters such as the number of interphase layers, "Y" and the same or different thickness of layers, the different levels for "E_i" are commonly calculated by these models. As a result, the Ji model is suggested to present an initial estimation for "E_i" in the polymer nanocomposites by tensile modulus.

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