Journal of Scientific & Industrial Research
Vol. 59, February 2000, pp 136-139

Non-isothermal Crystallization Kinetics of Glass Oil Palm Fibre Reinforced Phenolformaldehyde Composites

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Received: 7 June 1999; accepted: 13 December 1999

The non-isothermal crystallization kinetics of glass oil palm reinforced hybrid phenol formaldehyde composites has been studied at different heating rates by using Differential Scanning Calorimetry(DSC). Samples of different weight percentages of fibres have been tested and the results are analysed in the light of Ozawa's theory. It has been found that all the samples studied follow the above theory very well and, therefore, it has been employed to determine the Avrami exponent for these composites under non-isothermal conditions.

Introduction

A fibre hybrid composite consists of two or more fibres contained in a simple matrix[1]. Using the combination of two different fibers, a composite having desired properties can be fabricated. Thermo-setting resins are the preferred matrices for the composite fabrication. One of the most important thermo-setting polymer is phenolic resin. This resin is used as an industrial material because of its good heat resistance, electrical insulation,dimensional stability,and chemical resistance[2-4]. However, it is inherently brittle due to its higher cross-linking density. The properties of these resins can be modified by reinforcing it with different type of materials such as ceramics, elastomers, and fibres[5-7]. The usual reinforcing fibre used with thermo-setting resins for the manufacture of hybrid composites is the E-glass fibre. In this study resole type phenol formaldehyde(PF) resin matrix is reinforced with different weight percentages of two type of fibres (E-glass fibre and oil palm fibre), resulting in the hybrid composites. Method of preparation of these composites is already known[8-10]. Non-isothermal crystallization behaviour of polymers and composites is of great importance because most of the processing techniques occur under non-isothermal conditions. An attempt has been made to study crystallization kinetics of polymers, blends and composites under non-isothermal conditions[11]. Avrami exponent ,which renders information on the dimensionality of crystallization growth, is generally calculated under isothermal conditions. Here, an effort has been made to determine Avrami's exponent under non-isothermal conditions using Ozawa's [12] equation.

Materials and Methods

Differential Scanning Calorimeter(DSC) scans of all the composites were taken using Rigaku-8230 Model coupled to a Thermal Analysis Station(TAS). The temperature precision of the instrument is 0.1 K with an average standard error of about 1.0 K in the measured values. The samples between 10.0 and 20.0 mg were taken and scanned at the heating rates of 5,10,15 and 20 K/min.

Results and Discussion

The general form of the Ozawa's[12] equation is given by:

$$\ln[-\ln\{1-X(T)\}] = X - n \ln \alpha \,. \qquad ...(1)$$

where $[1-X(T)]$ is the untransformed volume fraction of the sample and is the heating rate. n is the Avrami exponent related to the type of nucleation and morphology.

The fraction crystallized at time t is calculated by the relation $X = A_T/A$, where A is the total area of the exotherm between the temperatures at which the crystallization begins and is completed. A_T is area between temperatures T_1 and T[13,14].

The value of $[1-X(T)]$,the untransformed amorphous fraction, is calculated as a function of temperature for different heating rates and are plotted in Figure 1 for sample No.1. Similar type of behaviour is found for the other composites. The value of amorphous fraction at a

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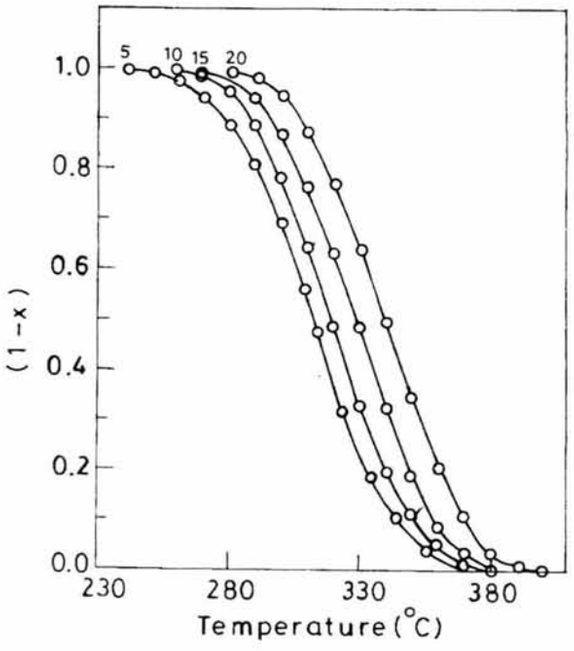

Figure 1 — Amorphous fraction as a function of temperature

given temperature is taken from these plots at different heating rates. Then the double logarithm of amorphous fraction ln[-ln{ 1-$X(T)$}] at constant temperature is plotted as a function of the heating rate. The curve of ln[-ln{ 1- $X(T)$}] vs ln is a straight line and follows the Ozawa's equation. The slope of the above curve gives the Avrami exponent and intercept determines the cooling crystallization function k . It can be observed from Figure 1 that the crystallization occurs at higher temperature corresponding to higher heating rate. Also, at each heating rate the crystallization takes place at the temperature below the point of maximum heating rate. This behaviour of the curves is suggestive of the fact that transformation is mainly controlled by nucleation process.

Figures 2(a) to 2(e) represent the plots of Ozawa's equation for samples 1,2,3,4 and 5 which are in good agreement with the experimental data. Figure 2 is also utilized for the determination of cooling crystallization function κ . The values of κ vary between 0.627 and 2.33 for these composites. The cooling crystallization function is independent of temperature. Avrami exponent, n, obtained from the slope of these curves for non-isothermal treatment is then plotted as a function of temperature in Figure 3. Values of Avrami exponent for

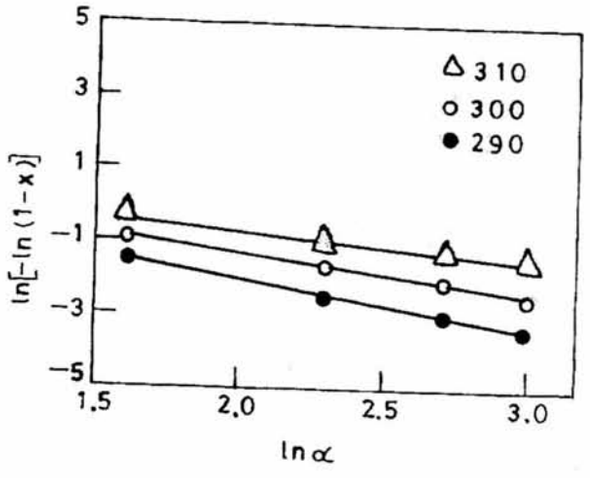

Figure 2(a) — Plot of ln[-ln(1-X)] vs ln α at different temperatures for composite No. 2

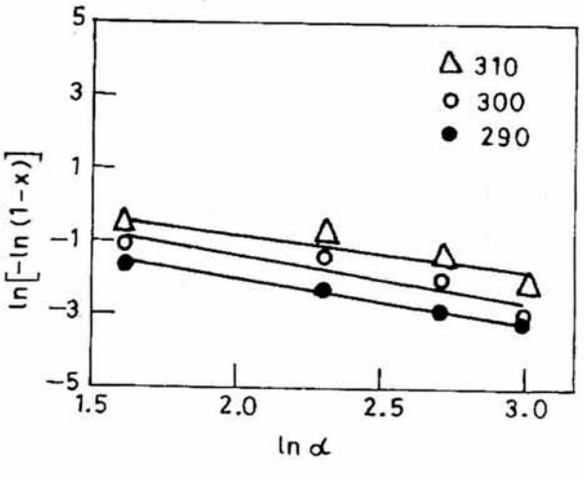

Figure 2(b) — Plot of ln[-ln(1-X)] vs ln α at different temperatures for composite No. 2

these composites are between 0.809 and 1.54. The Avrami exponent determined for these composites show a decreasing trend with the increase of temperature. This variation of n with temperature is attributed to the change in the nucleation mechanism. This variation of n has also been confirmed by optical microscopy for different polymers[15,16].

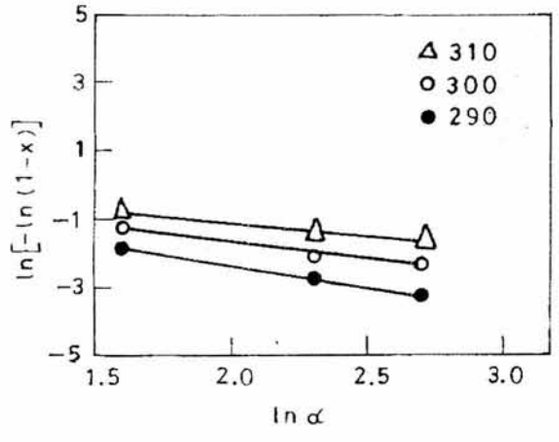

Figure 2(c) — Plot of ln[-ln(1-*X*)] vs ln α at different temperatures for composite No. 3

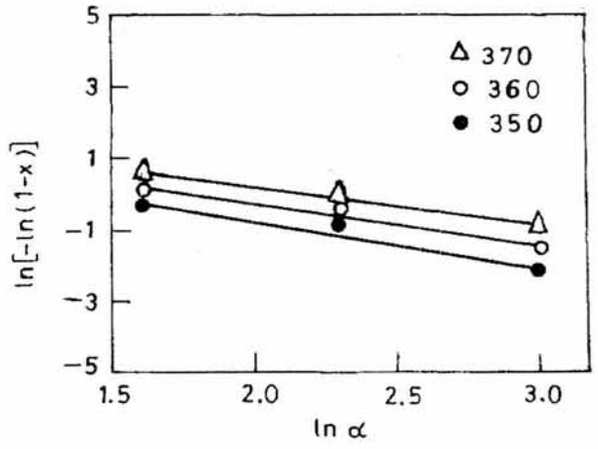

Figure 2(e) — Plot of ln[-ln(1-*X*)] vs ln α at different temperatures for composite No. 5

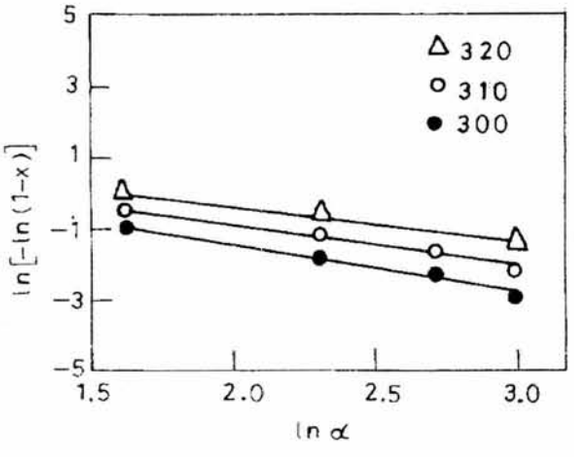

Figure 2(d) — Plot of ln[-ln(1-*X*)] vs ln α at different temperatures for composite No. 4

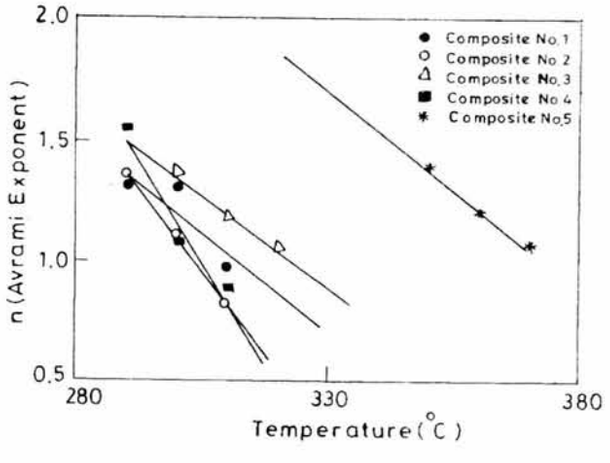

Figure 3 — Avrami exponent as a function of temperature

Conclusions

Application of the theory on the experimental results shows that Ozawa's theory can also be applied to polymer composites. This is an efficient theory to analyse the non-isothermal crystallization data.The equation fits very well in the experimental data and allows the determination of the Avrami's exponent. In general, Avrami's exponent,as determined by this method, is in good agreement with the values obtained by means of isothermal technique. Also, Ozawa's equation allows one to study the crystallization behaviour in wide temperature range. This indicates that the technique can be used as the complementary tool for the investigation of crystallization of polymers, as well as, composites.

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