X-RAY DIFFRACTION TECHNIQUE IN PROBING FILLERS IN THE POLYMER BLEND COMPOSITE

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ABSTRACT

We have illustrated the application of X-ray diffraction (XRD) and thermal measurements to meticulously investigate the positioning of nano-sized boron carbide (B_4C-type compounds) fillers. In this study, we successfully employed a deconvolution process using the Pearson VII function to fit the XRD diffraction Bragg and amorphous peaks, with a fitting error of below 4%. Additionally, thermal measurement was conducted by assessing the thermal conductivity of the composite material.

ABSTRAK

Kami telah menggambarkan aplikasi pembelauan sinar-X (XRD) dan ukuran terma untuk menyiasat dengan teliti kedudukan pengisi boron karbida (sebatian jenis B_4C) bersaiz nano. Dalam kajian ini, kami berjaya menggunakan proses dekonvolusi menggunakan fungsi Pearson VII agar sesuai dengan pembelauan XRD Bragg dan puncak amorf, dengan ralat pemasangan di bawah 4%. Selain itu, pengukuran terma dijalankan dengan menilai kekonduksian terma bahan komposit.

Keywords: X-ray diffraction (XRD), nano-sized boron carbide, thermal conductivity

INTRODUCTION

The location of nanoparticles as well as thermodynamic and/or kinetic effects affect several aspects of the morphology of polymer blends [1]. The assessment of wetting parameters, such as contact angle and interfacial tension, is necessary for the localization of nanoparticles in a polymer blend. However, because of the disruptions brought on by the filler's surface structure, it is challenging to determine precise values for the interfacial tension between the polymer and the filler [2]. Consequently, a fresh method for researching the nanoparticles' localization in polymer blends is required. The combination solution that has been suggested shows great promise because it is simple to use and doesn't require many computations. In order to determine the location of fillers in the polymer blend, two methodologies were combined to propose the combined method. Utilising X-ray diffraction (XRD) technology, the relative degree of crystallinity in the HDPE phase was tracked. The behaviour of heat transmission in the sample was observed using a thermal conductivity measurement technique. Fillers would cause the HDPE phase's crystallinity to be disrupted and facilitate heat transmission, particularly in the NR continuous phase. A smart decision was made to choose the right filler loading for the application by taking into account the fillers' behaviours in both the co-continuous and continuous phases as well as their contributions to the composite's overall qualities.

Studies using X-ray diffraction (XRD) usually entail phase analysis and crystallography of polycrystalline materials. Currently, semicrystalline materials, in particular HDPE, are being characterised using the same methodology. The distinctive peaks at 2 theta values of 21.43∘ for the (110) plane and 23.80∘ for the (200) plane [3] are what distinguish this material from others. These are the principal HDPE reflection planes. NR serves as a background in the study and can be seen as an amorphous halo in the diffractogram.

When assessing a composite material's thermal performance, thermal conductivity is important. Numerous research have looked into how filler size, shape, and type affect composites' thermal performance [4–8]. On the other hand, little information is available regarding the impact of fillers in the polymer blend. Assuming that the added filler is thermally more conductive than its surroundings, a rise in thermal conductivity was anticipated in this work once the filler was overly positioned in the continuous NR phase.

METHODOLOGY

Commercial grade H is provided by Titan Chemicals Sdn. Bhd. (Malaysia) and has a density of 0.961 g/cm3 and a melt flow index of 7 g/10 min. The Rubber Research Institute (Malaysia) provided technical grade NR, sometimes referred to as SMR L. This grade was used to create liquid natural rubber (LNR), which had a weight average molecular weight (Mw) of 4.8 × 105 in comparison to polystyrene. LNR was then employed as a compatibilizer in the blend. Nano-scaled boron carbide (Yemate Ind. Co. Limited, China) filler with an average size of 20 nm was utilised. LICA 38 from KenReact Co. Limited was the surfactant that was employed.

A polymer blend including 50% weight NR, 10% weight LNR, and 40% weight HDPE made up the matrix. Prior to the compounding stage, the treated fillers and HDPE were manually premixed for five minutes. The addition of the raw materials was done in three stages: NR, LNR, and then HDPE or premixed HDPE with fillers. The samples were combined for 15 minutes at 135 °C in an internal mixer, a Rheomix Haake 600P with roller-type rotors. The rotors were operating at 55 revolutions per minute (rpm). Before being characterised, the combined materials were heated and pressed to create samples.

RESULTS AND DISCUSSION

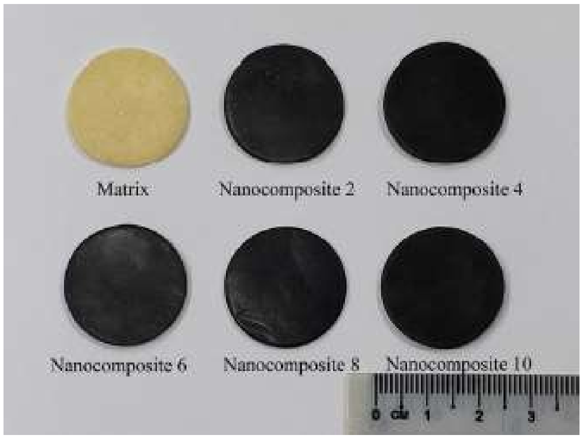

Figure 1. Samples

Figure 1 shows the samples. An example result of the deconvolution process is given in Figure 2, for a matrix sample. Fitting is considered successful in this work if the fitting error is less than 5%. The correlated degree of crystallinity trend for each sample with varying filler loadings is displayed in Figure 3. A comparison is made between the matrix and the nanocomposites' trends. The trend often indicates decreases in crystallinity with the addition of fillers relative to the matrix below 4%wt. Reduced HDPE crystallinity resulted from the filler taking up more space during solidification and upsetting the crystal structure. It's interesting to note that crystallinity decreases end around 4%wt. To the extent that it did not violate the minimum space required by HDPE to produce the HDPE crystals, the filler is allowed to remain in its phase by the HDPE phase. The trend of the rapid decrease in crystallinity up to a loading of 4%wt reflects this. The filler can readily reach the HDPE phase in this range.

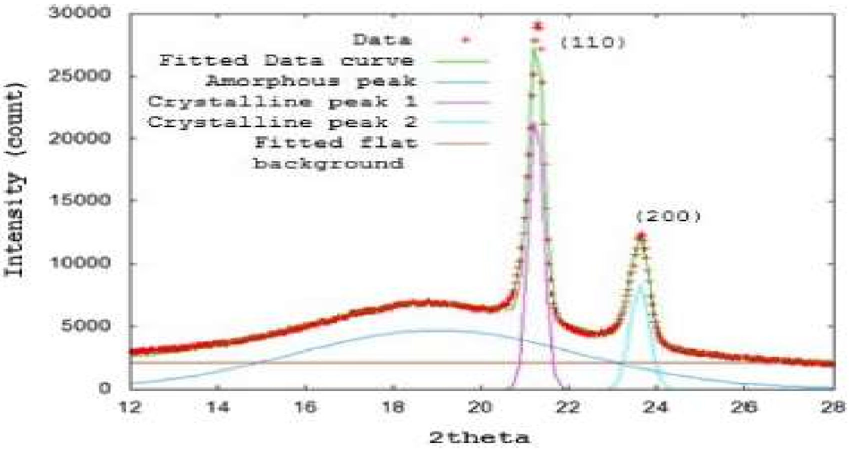

Figure 2. Deconvolution method

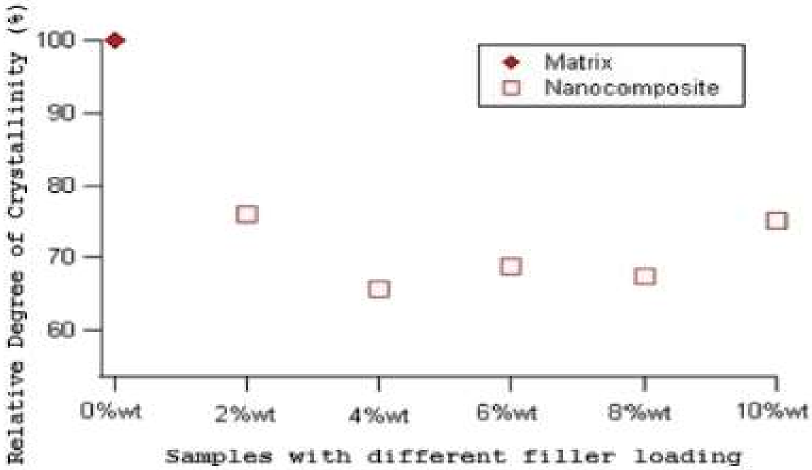

Figure 3: Results of the relative degree of crystallinity

The crystallinities suffered greatly as a result. There was no more reduction in crystallinity after this loading since the filler did not appear to be favourably present in the HDPE phase. The increase in thermal conductivity in Figure 4 indicates that the excess filler is expected to reside in the continuous NR phase. The crystallinity trend increases slightly from 4%wt to 10%wt of filler addition. The creation of filler agglomerates, modifications to HDPE morphology, and a poor premixing step could all be reasons for this since these could make it more difficult for the filler to readily occupy the HDPE phase. The impact of adding particle filler on the polymer's shape and crystallisation behaviour may provide the best explanation. This is crucial, particularly when the polymer is semicrystalline, and the filler concentration is minimal [9–11].

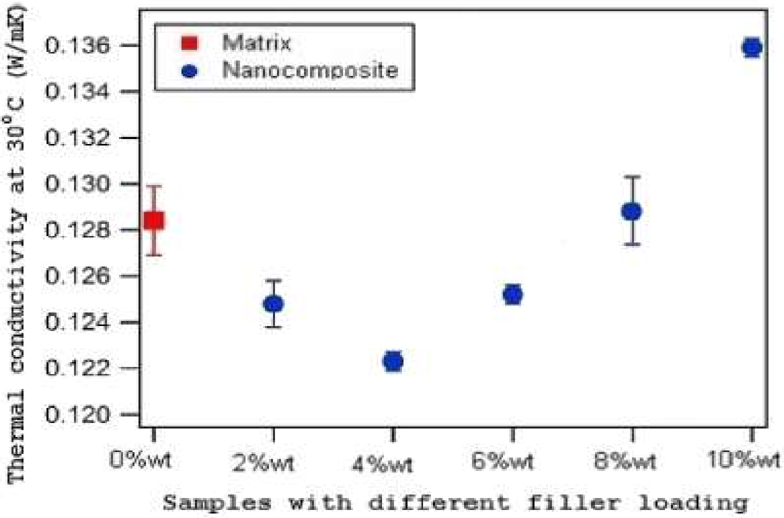

Figure 4. Results of thermal conductivity

CONCLUSIONS

In order to generate a well-tailored composite, it is imperative to monitor the filler's behaviour in both phases of the polymer mix to ensure optimal filler loading selection. The threshold was determined to be 4%wt of filler loading. The thermal conductivity was found to significantly rise above this amount. This was predominantly caused by the filler's behaviour, given it was mostly present in a continuous NR phase. Beyond 4%wt of filler addition, HDPE crystallinity increased significantly rather than decreased further. Thermal conductivity tests and XRD analysis are useful tools for tracking filler behaviour in polymer composites.

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