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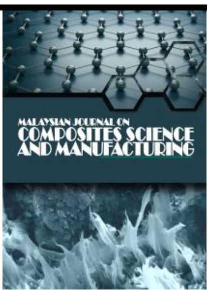

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Tensile Properties Degradation of Thermally Aged NR/EPDM Blend and Nanocomposites for Engine Mounting

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ABSTRACT

Nowadays, NR/EPDM have drawn great attention to most researchers to be utilized in an engine mounting. In service, majority of structures and components are subjected to service condition such as heat, liquids and stress of dynamic loading. This research is an effort to compare the thermal effects on physical and mechanical properties of NR/EPDM blend and NR/EPDM filled graphene nanoplatelets (GNPs) for the potential usage in the engine mounting. The blends and nanocomposites were prepared through a melt compounding using a Banbury Internal Mixer, exposed to thermal cycles and tensile tested. The materials were heated alternately for 10 minutes at 60℃ and 120℃ for 0, 35, 70 and 150 cycles. Thermally aged NR/EPDM nanocomposites showed higher performance under tensile stress if compared to NR/EPDM blend. The tensile strength and elastic modulus were consistently 40% higher than the strength of the blend. This is due to the reinforcing effects of GNPs and their good interaction with the matrix. The reduction of tensile properties was due to the embrittlement effect and in line with the increase in amorphous phases from the broadening of XRD spectra. This has proven that the NR/EPDM nanocomposites can withstand thermal cycles, oils and fluctuating stress better than NR/EPDM blend and exhibited higher potential to be utilized as the engine mount material.

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1. Introduction

Engine mounts are critical component in passenger cars. Its function is to isolate the vibration from an engine to car body. They are two categories of engine mount; passive engine mount and active engine mount. Mostly passive engine mount is made from elastomeric materials either, natural or synthetic rubber or the blend of the rubber. It absorbs the engine vibrations and road shocks to dampen the noise and vibration for comfort. It is sensitive to temperature [1] hence the life span and performance of an engine mount affected by different temperatures acting on it. However, the change in mounting criteria is highly contributed by the elastomeric component inside the mount [2]. Therefore, understanding the effect of materials structure on the thermal ageing behavior is crucial for engineers and designers to improve the system.

Natural rubber (NR) is a biopolymer with high resilience, superior elasticity, high fatigue resistance, low level of strain sensitivity, and great processability [3]. However, NR weak upon various environmental factors such as humidity, light, ozone, radiation and moisture due to chemically reactive of highly unsaturated NR structure. In contrast, ethylene-propylene-diene (EPDM) exhibits excellent ageing properties due to saturated carbon-hydrogen polyolefinic rubber including good thermal, ozone and chemical resistances. The platelet thickness of graphene nanoplatelets (GNPs) is in the range of 0.3 – 100 nm and high aspect ratio combined with the extraordinary properties make GNPs ideal reinforcing fillers in polymer nanocomposites [3]. It combines the superior criteria of carbon nanotubes and layered silicates. Furthermore, GNPs are proven to enhance the properties of various polymer nanocomposites hence considered as the most versatile functional and filler reinforcing along with economical graphite sources [4].

There are numerous number of studies carried out to improve the properties of engine mounting to isolate the engine vibration, optimizing the engine performance, extending the engine's life span and saving power consumption [2, 5-6]. Our previous studies on GNPs reinforced NR/EPDM blends proven to increase the heat dissipation [4] and improve vibration damping [7]; reflecting their potential to be used in the engine mounting. Unfortunately, there are currently no available research investigating the effect of thermal aging on tensile properties of both NR/EPDM blends and nanocomposites. It is crucial since there are constant tensile stresses and temperature gradients experienced by the mounting system during service from the weight of the engine and heat transfer during the engine run. According to Sathawane and Patil [8], the temperature range experiences by the mount are -30 °C to 120 °C. However, the service temperature may be accidentally exceeded the nominal parameters and causing the materials to experience multi-stress degradation. Thus, a structural alteration in the heat capacity of rubber components via incorporation of thermally conductive GNPs may improve the heat transfer as well as heat dissipation rate of the mount system [9]. As the results, it may prolong the life span and enhance the performance of the component.

This study is to investigate the thermal effect and compare the tensile properties of thermally aged NR/EPDM blends and NR/EPDM nanocomposites at two different temperatures; 60 °C and 120 °C under the influence of various thermal cycles.

2. Methodology

2.1 Materials

The natural rubber, SMR 20 grade and epoxidized natural rubber were supplied by Lembaga Getah Malaysia whereas, EPDM Buna® EPT 9650 was procured from Lanxess Corporation. The SMR 20 has low viscosity of 80 ± 10 and ash content and volatile matter of 1.0 and 0.8 %wt, respectively. Meanwhile, the EPDM has mooney viscosity of 60 ± 6 and ethanol as well as ENB content of 53 ± 4 and 6.5 ± 1.1, respectively. Graphene Nanoplatelets (GNPs) KNG-G2 was supplied by Xiamen Graphene Technology Co. td, China. The thickness and diameter of GNPs KNG-G2 are < 2 nm with less than 3 layers and 5-20 µm individually with carbon content of >99.5 wt%. Other compounding ingredients such as zinc oxide, stearic acid, and sulphur were acquired from System/Classic Chemical Sdn Bhd., Perkacit-MBTS and Perkacit-TMTD were purchased from Perkacit whereas the 6PPD was supplied by Flexsys America (United States).

2.2 Sample Preparation, Testing and Analyses

Both NR/EPDM blends and nanocomposites were synthesized via melt compounding according to ASTM D 3192 using Banbury BR 1600 internal mixer. The compounding formulation (Table 1) used was semi-efficient vulcanization (semi-EV) system [10]. About 1.12 kg at one time was mixed with fill factor of 0.7. Both NR and EPDM were first masticated at 70 °C for 10 minutes using internal mixer at rotor speed of 110 rpm prior to rubber blend preparation. Firstly, NR, EPDM and ENR-50 were masticated for 1 minute before added with the remaining ingredients except sulphur and accelerators. Finally, sulphur and accelerators were added and mixed for 1 minute before the mixture was dumped and cooled to room temperature. Then, the blend and nanocomposites were hot pressed into 2 mm sheets using GT7014-A from Gotech at 150°C for the respective cure times, t90 derived from a rheometer test in accordance with ASTM D 2084. Next, the samples were cut into 64 mm x 10 mm dog bone sample sizes. Then the samples were thermally aged at 60°C and 120C for 0, 5, 35, 70, and 150 times alternately within 10 minutes. The nomenclature used for the blend and nanocomposites are NR/EPDM blend X-Y and NR/EPDM nanocomposites X-Y followed by the X aging temperature and Y thermal cycles. Then, it was subjected to tensile test according to ASTM D1822 using Universal Testing Machine (Toyoseiki Strograph). The testing was held at ambient temperature and at a cross-head speed of 500 mm/min. A minimum of seven samples was tested per every set of experiments to make sure a high confidence level.

Table 1
Compounding formulation for NR/EPDM blends

Ingredients	NR/EPDM Blend (phr)*	NR/EPDM Nanocomposite (phr)*
NR (SMR 20)	70	70
EPDM	30	30
ENR-50	10	10
Zinc oxide	5.0	5.0
Stearic acid	2.0	2.0
Sulphur	1.5	1.5
MBTS[a]	1.0	1.0
TMTD[b]	0.3	0.3
6PPD[c]	2.0	2.0
GNPs (wt%)	0.0	3.0

*Parts per hundred rubber
a 2,20-dithiobis (benzothiazole)
b Tetramethylthiuram disulphide
c N-(1,3-Dimethylbutyl)-N’-phenyl-p phenylenediamine

Then, the fracture surfaces were observed under secondary electron imaging mode using a scanning electron microscopy, model EVO-50 from Zeiss at 500X magnification. Prior to the examination, the samples were placed onto aluminium stubs and sputter coated with conductive gold layer. In addition, X-ray diffractometry (XRD) analysis was performed utilizing a PANalytical X'Pert PRO diffractometer and characterized using CuKα radiation with λ = 1.5418 nm under conditions of 2θ scanning range at 5° to 90°. Besides, the compositional analysis was performed using a JASCO FTIR 6100 by the attenuated total reflectance infrared (ATR/IR) spectroscopy at the resolution of 4 cm^{-1} at the scanning range of 500-4000 cm^{-1}.

3. Results & Discussion

3.1 Tensile Strength

In Figure 1, the tensile strength for both blends and nanocomposites showed an identical trend as the number of thermal cycles increases. The tensile strength of samples decreased as the number of thermal cycles increased. Both materials was observed to manifest a rather dramatic change in the tensile strength at 60°C (Figure 2a). On the other hand, a drastic drop of about 70% was observed for both blends and nanocomposites exposed to the thermal cycles of 35 cycles at 120 °C. This may due to the embrittlement mechanism that have taken placed inside the materials due to the stored heat with the increasing thermal cycles. This was supported by smoother tensile fracture surface of NR/EPDM blend, which indicated less ductile fracture behaviour. This was due to the chain embrittlement of NR/EPDM molecules at elevated temperature, which transformed the material to glassy state hence exhibit brittle-like failure under stress.

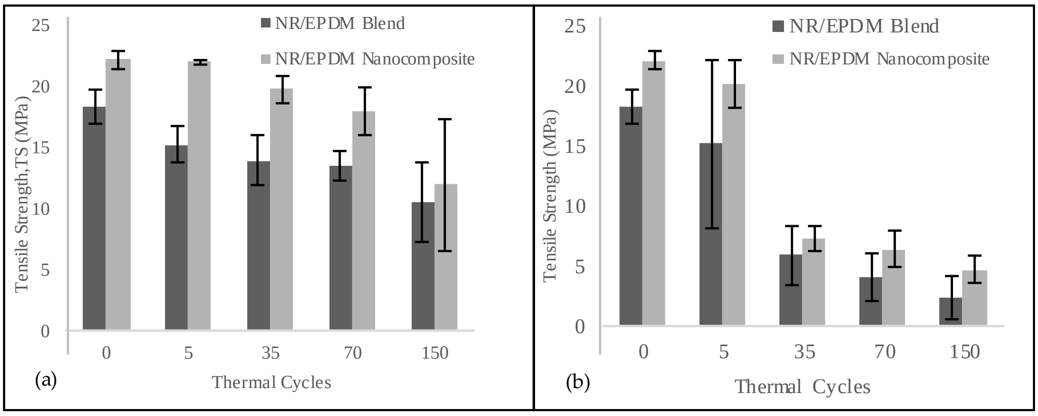

Fig. 1. Effect of thermal cycles at 60 °C and 120 °C on the tensile strength of NR/EPDM blends and NR/EPDM nanocomposites

However, the nanocomposites were observed to retain higher tensile strength of about 20 to 40 % compared to blends consistently; even at lower or higher thermal cycles. This is due to the higher specific surface area of nanofiller that is in contact with rubber phase, the higher stress transfer have taken place at the interface between the rubber phase and nanofiller. This is in agreement with Bokobza (2009) [11] in his work on carbon based reinforced elastomer; it reveals that the addition of carbon based nanofillers lead to an increase in its tensile properties. Furthermore, the improvements observed are higher than provided by the conventional filler such as carbon black. This observation is in line with the XRD results. The intensity of the peaks dropped significantly at the higher temperature (120°C) thermal cycles compared to the lower temperature (60°C) thermal cycles.

3.2 Modulus of elongation at 100% (M100)

Modulus is a common method to measure the stiffness of a material. A high value of tensile modulus means a high level of stress is required to produce a given strain. Figure 2 shows the tensile modulus of NR/EPDM blend at 100%. The M100 constantly increases with the increase of thermal cycles except of trivial changes for the blends exposed to 60°C. It is well accepted that modulus of a material is directly correlated to its stiffness. Therefore, this trend indicates that the stiffness of rubber blend raises with the increase of thermal cycles. Increasing stiffness could be associated to the post crosslinking as well as chain embrittlement of the rubber due to thermo-oxidation.

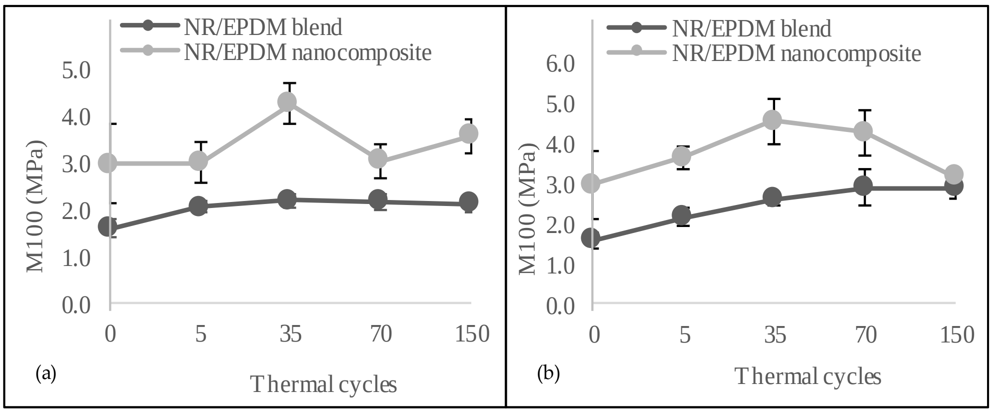

Fig. 2. Effect of thermal cycles at (a) 60°C and (b) 120°C on the modulus at 100% elongation (M100) of NR/EPDM blends and NR/EPDM nanocomposites

For NR/EPDM blends exposed to thermal cycles at 60 °C, the M100 values showed trivial changes due to lesser restriction of molecular motion by the fillers. In contrast, blends exposed to higher temperature at 120°C showing constant increment in M100. It experienced higher chain embrittlement level due to thermo-oxidative aging if compared to blend at 60°C. For the nanocomposites, modulus increases during the early stage and then decreases with further increasing thermal cycles. The increment in modulus at low thermal cycles may be due to the post crosslinking. While, the reduction in the modulus after this increment may be attributed to the softening effect due to chain scissoring [5] or degradation of graphite due to thermo-oxidation; as observed in XRD analyses. The chain's weakening mechanism due to the embrittlement diminish the structural integrity of the composites resulted in cut-off into shorter polymeric chains. Meanwhile, later increase in M100 in the nanocomposites subjected to aging at 60°C increase in the modulus after this slight decrease can be attributed to the chain cleavage mechanism. The generated free radicals will from new crosslinks which later resulting in an overall increase in the crosslink density. Overall, the reason for the general increase in the modulus attributed to the crosslinking reactions outweighing the softening mechanism in nanocomposites, especially for the nanocomposite at 60°C [5].

3.3 Elongation at break (EB)

Figure 3 shows the EB decreases with the increase of thermal cycles. The changes are almost stationary with the raise of the number of thermal cycle for the blends and nanocomposites exposed to 60 °C. But, rather drastic changes were observed in both composites subjected to 120 °C. This observation is almost identical with the decrease in tensile strength at 120 °C. The EB for both blends and nanocomposites at 120°C were drastically reduced as the thermal cycles exceeded 35 cycles.

This in an indicator that 35 thermal cycles is the threshold point for the NR/EPDM blends to maintain structural integrity due to the thermal cycles effect at high temperature. This is explained by the morphological observation. It is noted that higher temperature thermal cycles accelerate the chains embrittlement and premature breakage under stresses hence increase the tendency for brittle failure to the material.

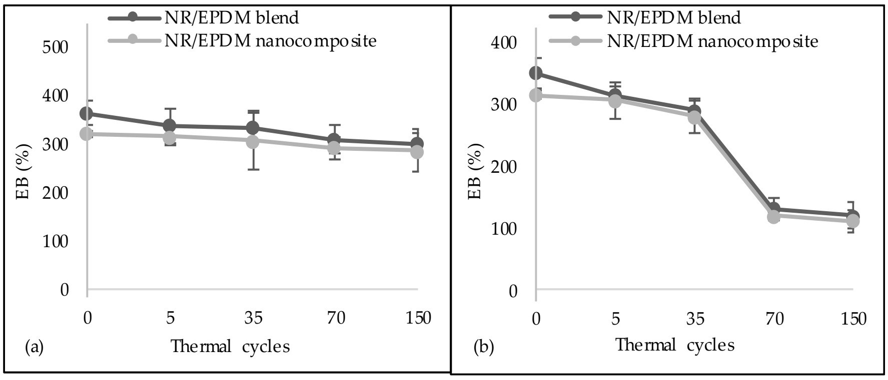

Fig. 3. Effect of thermal cycles at 60 °C and 120 °C on the elongation at break (EB) of NR/EPDM blends and NR/EPDM nanocomposites

3.4 Correlation between EB and M100)

Figure 4 depicts the pattern of correlation between EB and M100 plotted using log function for both NR/EPDM blends and NR/EPDM nanocomposites under thermal cycles at 60 °C and 120 °C temperatures. There are three apparent patterns prevailed from the thermal aging effects which labelled as Type I, Type II and Type III in Figure 4. The aging patterns observed are in agreement with the Type I, Type II and Type III aging mechanism according to the Ahagon plot [12-13].

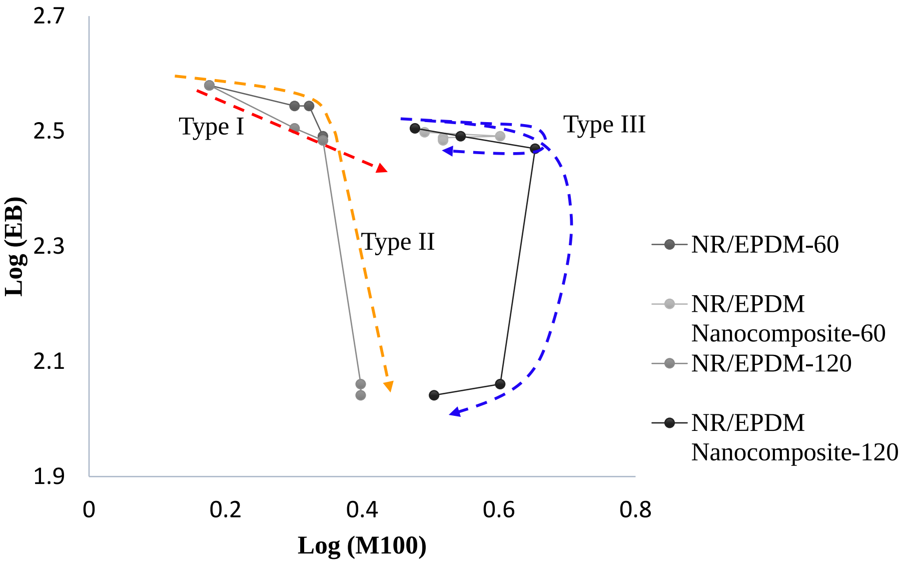

Fig. 4. Elongation at break (EB) versus Modulus at 100% (M100) of NR/EPDM blends and NR/EPDM nanocomposites at 60 °C and 120 °C thermal cycles

In the Type I, the highest negative gradient indicates there was constant increased in M100 and decreased in EB as increased in the number of thermal cycles or total accumulated temperature exposure time (TATET). It is noted that the negative gradient for linear portions of the nanocomposites were lower than the blends, which represents slower degradation rates at this point. The NR/EPDM blend at low thermal cycles exhibited Type I thermal aging criteria [5] since the M100 increased and EB decreased gradually as increased in thermal cycles; the Type 1 is post-crosslinking predominance at lower temperature (60 °C) thermal cycles. Furthermore, the Type II model indicated a drastic dropped at approximate 60% in EB and a very little increased in M100 with an increased in thermal cycles at 120 °C. It showed prominence effect of chain embrittlement, which accelerates the brittle failure of the blends, good agreement with the fracture surface. Meanwhile, for the NR/EPDM nanocomposites, the samples manifested the Type III aging mechanism. It is obvious that the nanocomposites exhibited softening effects once it exposed to the thermal cycles at about 35 cycles due to chain scission mechanism. Then, followed by chain cleavage mechanism at higher cycles, which contributed to the higher total crosslinks formation. In overall, the M100 of NR/EPDM nanocomposites were higher than NR/EPDM blend samples nearly 60% and 30% at 60 °C and 120 °C, respectively. The indication depicted the presence of GNPs helps to extent the strength of rubber by increase the interaction between matrix and filler. The M100 of the nanocomposites remained higher than the control samples even at high temperature of 120 °C.

3.5 X-Ray Diffraction Analysis (XRD)

The position and intensities of X-ray diffraction peaks could be utilized to analysis the effect of thermal cycles on the phase identification and level of crystallinity in both NR/EPDM blends and nanocomposites. Figure 5 depicts the XRD diffractogram for vulcanized NR/EPDM blends and nanocomposites.

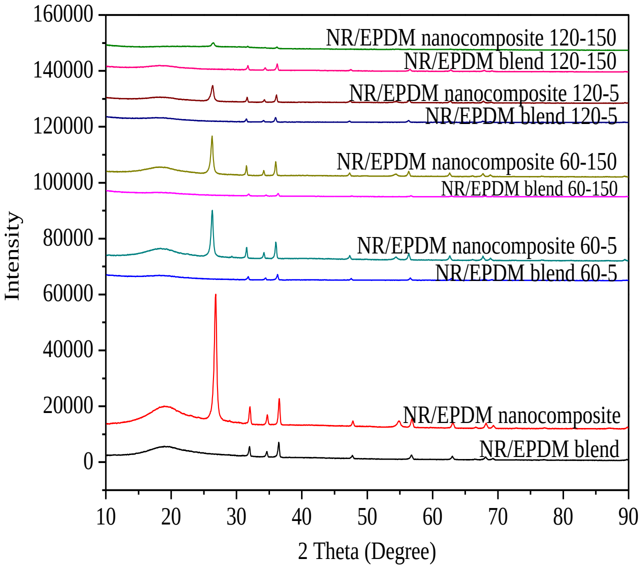

Fig. 5. XRD spectra of NR/EPDM blends and nanocomposites at 60 °C and 120°C thermal cycles

Broadening diffraction peak indicates the amorphous structure [7] whereas sharp peaks indicate the crystalline regions. It is observed for the control samples that the blends presented lower crystallinity than nanocomposite hence lesser sharp peaks were observed. The nanocomposite showed a sharp reflection at 2θ = 26.6° which represents the presence of graphene nanopletlets in the materials [14]. The agglomerated graphene nanoplatelets manifested as graphite in the composites. Meanwhile, there is none sharp peaks presence in any of the NR/EPDM blend. When the thermal cycles were introduced to the NR/EPDM blends and nanocomposites, the intensity of the peak was gradually dropped to almost 90% as the temperature and thermal cycles increased. This trend suggests the development of amorphous phase in the materials, which certainly resulting from the thermo-oxidative aging. The peak broadens and the sharpness reduces due to the transformation of used-to-be crystalline structure to amorphous structure. This is especially observed to take place in the graphite phases of the NR/EPDM nanocomposites since the weak van der Waals which holding the graphite sheets together is overcome. Then, the graphite transformed to numerous single layer of graphene sheets in the matrix. The heat energy from the thermal cycles would attack the weak Van der Waals force between the graphene sheets layers rather than the stronger C-C covalent bond of the graphene. Meanwhile, the reduction of peak at $2\theta = 34.3°$ in the XRD spectra may be originated from the degradation of (002) plane of ZnO [10], from the thermal cycles effect.

3.6 Fourier Transform Infrared Spectroscopy (FTIR)

Attenuated total reflectance infrared (ATR/IR) spectroscopy was carried out to identify the functional groups present in the materials. Figure 6 shows IR spectra of NR/EPDM blend and nanocomposite comparing the effect of the highest thermal cycles of 150 at 120 °C. In overall, the spectra of NR/EPDM blend and nanocomposites under thermal effect were almost identical. It showed dominant peaks at 2950 and 2853 cm^{-1} which corresponded to the C–H vibrations of methylene group vibrations. This is in good agreement with the report by Gunasekaran et al. (2007) [14]. Meanwhile, the bands around 1445 cm−1 are assigned to -CH2 scissoring vibrations from the propylene unit. However, comparison of the IR spectra of thermally aged with the neat NR/EPDM blend and nanocomposites presented in our previous work in Yaakub [15] showed the increment of intensity at which corresponds to the C-S of the CS2 from the post-crosslinking which taken placed due to the thermal aging.

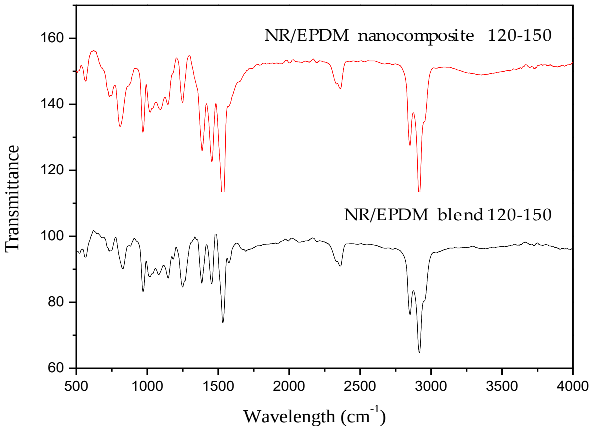

Fig. 6. FTIR graph of NR/EPDM blends and nanocomposites at 120 °C for 150 thermal cycles

3.6 Morphological analysis

Figure 7 illustrates the SEM micrograph taken from the tensile fractured surfaces of NR/EPDM blends and nanocomposites comparing the slightest and the worst thermally aged composites. In both condition, the nanocomposites, which exhibited higher tensile strength than blends showing morphology of relatively rougher surfaces with apparent shear yielding mechanism, especially for the one subjected to 60 °C. Meanwhile, the blend samples with relatively lower tensile strength showed less tearing line. In this case, the rubber matrix experienced rather less plastic deformations from shear yielding; smoother surface represents increment in brittle-like failure mode. The nanocomposites manifested a higher level of ductile failure due to the good interaction between GNPs and matrix, which demonstrated in the higher tensile properties than blends at particular thermal effect parameter. The tearing line thickness reduces with the increase in both temperature and thermal cycles that represented by Figure 7 (c) and (d) if compared to the surfaces at 60 °C and 5 thermal cycles. This is due to the increase in brittle-like failure from the effect of thermo-oxidative aging. The changes in morphological characteristics at low and high level thermal aging are the results of crosslinks density and degradation level experienced by the rubber molecular chain [17].

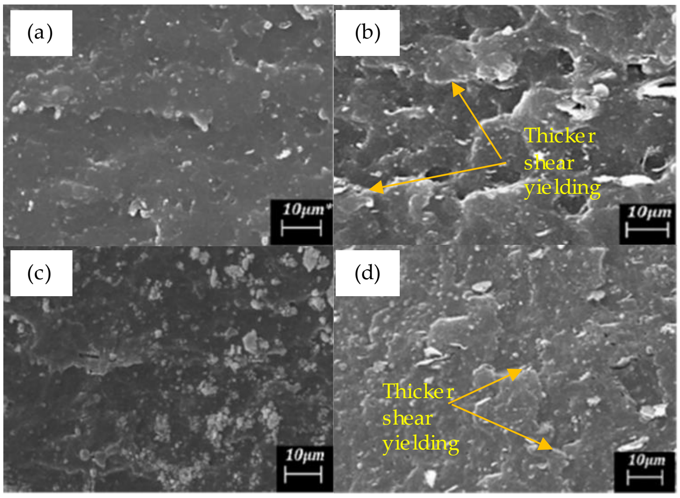

Fig. 7. SEM micrographs of the tensile fracture surface of NR/EPDM blends and nanocomposites at 500X magnification. (a) NR/EPDM blend 60-5; (b) NR/EPDM nanocomposite 60-5; (c) NR/EPDM blend 120-150 and (d) NR/EPDM nanocomposite 120-150

4. Conclusions

In this study, the NR/EPDM blends and nanocomposites have been thermally aged at 60 °C and 120 °C for the 0, 5, 35, 70 and 150 cycles. Three types of thermal aging behaviours (Type I, II and III) were predominant from the M100 and EB interaction. These attributed by the embrittlement and softening effects due to post-crosslinking as well as chain scissoring mechanisms. The morphological analyses depicted the aged blends especially at higher temperature with the increment in thermal cycles resulted in more brittle-like failure. In oppose, the nanocomposites exhibited thicker matrix shear yield and stronger interface which known as ductile fracture surface. The presence of GNPs played an important role to enhance the interaction between rubber-filler for the improvement of thermal aging properties of NR/EPDM. Therefore, NR/EPDM nanocomposites indicated a promising potential to enhance the tensile properties and thermal stability of engine mounting.

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