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Research Article

Influence Calcium Carbonate Nano-particles $CaCO_3$ on Crack Growth Fatigue for NR Compound

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Abstract

This The aim of this work was to study the influence of Calcium Carbonate CaCO3 nano-particles (N-CC) in preparation NR compound . Four compositions of NR/ CaCO3 nanocomposites with filler loading Calcium Carbonate of [0, 3, 5, 7 and 10] hpr, the compositions were prepared in Laboratories Inc. Tires Babylon*. To determine the crack growth fatigue at test device called (Me Mattia device) was used According to [ASTM D813], operating at a constant speed under load and giving 300 flexing cpm. The test included the Crack growth Fatigue properties of NR/ CaCO3 nanocomposites compound using a vulcanization system [ASTM D3182]. The Material of filler used in this research is Carbon black (N326) with quantity constant (50 pphr). The crack growth fatigue test shown that the crack length is decreased with the increasing of percentages of CaCO3 nanoparticles.*

Keywords: *Calcium Carbonate CaCO3 nanoparticles (N-CC), filler loading, NR compound,* Crack growth Fatigue*.*

1. Introduction

Recently, CaCO3 nanoparticles were commercially available for toughening polymers. Although small and uniform particles are believed to be more effective in toughening than large ones, nano-particles, even if they were surface-modified, often agglomerate and cause a significant decrease in toughness. Hence, failure to obtain uniform nanoparticles dispersion in the polymer matrix is a first major challenge for effective toughening. So thought that the interfacial interaction between nanoparticles and polymer matrix is one of the most important factors affecting the crystalline morphology of the nanocomposites(Weitao Wan *et al*, 2006).

On other hand, Calcium carbonate nanoparticles have been widely used as fillers in polymeric materials with the main purpose of reducing costs (Weitao Wan *et al*, 2006; Tjong S.C., 2006), however recently works showed that the incorporation of calcium carbonate nanoparticles can lead to higher impact resistance associated with higher elastic modulus (Thio YS *et al*,2002; Chan CH *et al*, 2002; Lin Y *et al*, 2005).

In 2009, Eiras and Pessan studied the influence of calcium carbonate nanoparticles in crystallization process of polypropylene. Four compositions of PP/CaCO3 nanocomposites were prepared in a co-rotational twin screw extruder machine with calcium carbonate content of 3, 5, 7 and 10 wt. (%). The tests included SEM analyzes for calcium carbonate, differential scanning calorimetry (DSC) and wide angle X-ray diffraction (WAXD) for the nanocomposites. The results showed an increase in PP crystallization temperature and crystallinity degree, and a reduction in spherullites size.

In 2006, Wan , yul, Xie, Guo, Mao and Huang (Weitao Wan *et al*, 2006) sdutied the effects of the surface-treatment of CaCO3 nanoparticle on the crystalline morphology of polypropylene. The dielectric properties of the nanocomposites were tested using the dielectric analyzer (DEA) at room temperature.

S. Koh, A. Saxena, W. D. van Driel, Zhang, R. Tummala In this research, numerical analysis has been employed to study the semi-elliptical crack growth and shape evolution in nanostructured interconnects subject to uniaxial fatigue loading. The results indicate that nanocrystalline copper is in fact a suitable candidate for ultra-fme pitch interconnects applications. . This study also predicts that crack growth is a relatively small portion of the total fatigue life of interconnects under LCF conditions. Hence, crack initiation life is the main factor in determining the fatigue life of interconnects.

Nowadays, the improvements of mechanical and chemical properties of material with addition of nano-sized filler are widely done. The introduction of nanofiller in the structure of a composite can develop the interaction strength between the polymer matrix and nanometric fillers, which can increase significantly

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the electrical, thermal, and mechanical properties (T. Tanaka, 2005; A. Motori, 2005). As an example, the MMT nanofiller are often used as an enhancement of composite tensile properties (Z. Jin *et al*, 2011).

2. Experimental work

2.1 Materials

The Elastomers used in this study, i.e. NR (Nature Rubber RSS) was supplied by the laboratories of the public company of tires Babylon. Carbon black (N330) and other fillers Materials was obtained from same company too.

The calcium carbonate Nanoparticles size 50 nm and Specific surface 20m^2/g produced in China. The four loading of filler calcium carbonate $CaCo_3$ nanoparticles of [0, 3, 5, 7, 10] hpr.

2.2 Preparation Specimens

Rubber specimens for various mechanical testings, including the tensile test, hardness test, density test, were prepared by mixing the rubber compounds. The formulation of rubber compounds was shown in Table 1

Table 1 Compound Recipes

Ingredient	NR blend (hpr)
NR (RSS)	100
Zinc oxide	5
Streaic Aid	2
Fillers1	variable 1
P-oil	4
6PPD(Antioxidant)2	3.25
Sulphur	1.8
MBS3	0.8

[1] According to Table 2
[2]N-(1,3-dimethylbutyl)-N´-phenyl-p-phenylene diamine
[3] 2-(4-morpholinothio)benzothiazole

Table 2 Fillers loading

Compound Recipes	Carbon black (N-326)	Nano-calcium carbonate
NR/N-CC0	50	0
NR/N-CC3	50	3
NR/N-CC5	50	5
NR/N-CC7	50	7
NR/N-CC10	50	10

2.3 Determination of Mechanical Properties

2.3.1 The fatigue device has an adjustable stationary head, or member provided with suitable grips for holding one end of each of the test specimens in a fixed position, and a similar reciprocating member for holding the other end of each specimen. The reciprocating member is so mounted such that its motion is straight in the direction of and in the same plane as the center line between the grips. The travel of the moving member is adjustable and is obtained by means of a connecting rod and eccentric having a minimum length ratio of 10 to 1. The eccentric is driven by a motor operating at a constant speed under load and giving 300 flexing cpm. Fig. (1) shown fatigue the device.

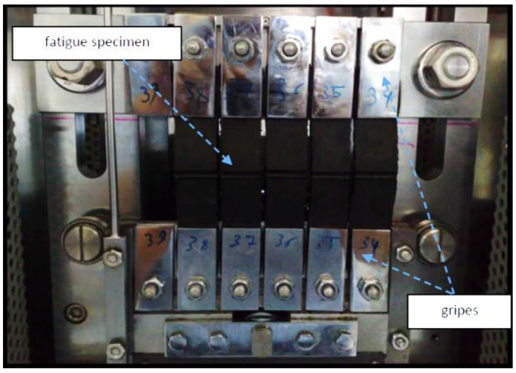

Fig. (1) De Mattia tester with time switch for starting or stopping, arranged with specimens for Flex-Cracking Test.

2.3.2 The test specimens [fatigue (crack growth) test ASTM D 813] were compression molded 350 bars at 160°C, and cure time 20 min, [ASTM D 3182].

3. Crack Growth Fatigue Theory (Paris' law of Elastomer Materials).

Paris' law relates the stress intensity factor range to sub-critical crack growth under a fatigue stress regime. As such, it is the most popular fatigue crack growth model used in materials science and fracture mechanics. The basic formula reads.

$$\frac{da}{dN} = C\Delta K^m$$

Where a is the crack length and N is the number of load cycles. Thus, the term on the left side, known as the crack growth rate, (M. Ciavarella *et al*, 2005) denotes the infinitesimal crack length growth per increasing number of load cycles. On the right hand side, C and m are material constants, and ΔK is the range of the stress intensity factor, i.e., the difference between the stress intensity factor at maximum and minimum loading (Roylance, *et al*, 2001). This led to the definition of three regions in da/dN-ΔK diagrams, namely zones I, II, and III.

Crack Propagation Stages.

Fatigue crack propagation can be divided into three stages as shown in Fig. (2): crack initiation I (CI), stable crack growth II (SCG), and unstable crack growth III (UCG).

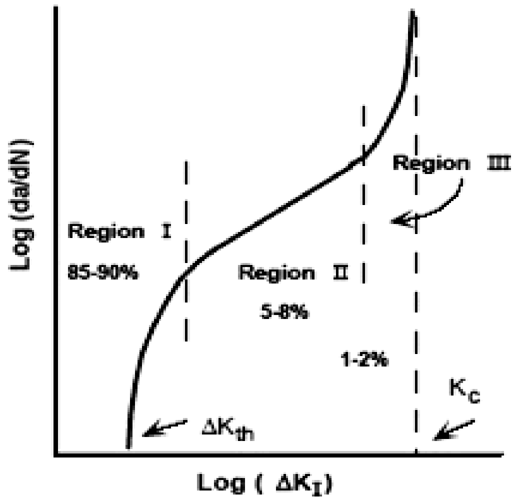

Fig. (2) The Paris law for fatigue crack growth rates.

Paris' law can be used to quantify the residual life (in terms of load cycles) of a specimen given a particular crack size. Defining the crack intensity factor as

$$K = \sigma\sqrt{\pi a}$$

Where σ is a unifor tensile stress perpendicular to the crack plane and Y is a dimensionless parameter that depends on the geometry, the range of the stress intensity factor follows as

$$\Delta K = \Delta\sigma\sqrt{\pi a}$$

where Δσ is the range of cyclic stress amplitude. Y takes the value 1 for a center crack in an infinite sheet. The remaining cycles can be found by substituting this equation in the Paris law

$$\frac{da}{dN} = C\Delta K^m = C(\Delta\sigma\sqrt{\pi a})^m$$

For relatively short cracks, Y can be assumed as independent of a and the differential equation can be solved via separation of variables

$$\int_0^{N_f} dN = \int_{a_i}^{a_c} \frac{da}{C(\Delta\sigma\sqrt{\pi a})^m} = \frac{1}{C(\Delta\sigma\sqrt{\pi a})^m} \int_{a_i}^{a_c} a^{-\frac{m}{2}}\, da$$

where N_f is the remaining number of cycles to fracture, a_c is the critical crack length at which instantaneous fracture will occur, and $a_i$is the initial crack length at which fatigue crack growth starts for the given stress range Δσ. If Y strongly depends on a, numerical methods might be required to find reasonable solutions.

For the application to adhesive joints in composites, it is more useful to express the Paris Law in terms of fracture energy rather than stress intensity factors (Wahab *et al,* 2005).

4. Results and Discussion

4.1 The Experimental Results of Fatigue (Crack Growth) Test

Me Mattia device was used to test the samples that had been prepared according to the Standard [ASTM D813]. The specimens were tested for 5 samples of the NR/N-CC compound used in this research, and at room temperature, where the results obtained are as shown in the following table (3):

Table (3) Results of crack growth test for NR compound, and the ratio of N-CC

Number of Cycles	Crack of Length (mm)				
	N-CC0	N-CC3	N-CC5	N-CC7	N-CC10
1000	3	3	3	2.5	2.5
3000	3.5	3.5	3.5	3	3
5000	4	4	3.5	3.25	3.25
7000	4.5	4.5	4	3.5	3.25
9000	5	5	4.5	4	3.5
11000	5.5	5.5	4.75	4.25	4
13000	5.75	5.5	5	4.5	4.5
15000	6	5.5	5.25	5	5

4.2 The crack length against number of cycles

Figure (3) show the values of the crack growth test to the vulcanized rubber and refers to the decreased crack length when the number of cycles and the percentages of the N-CC are increasing. This means that a crack does not grow more N-CC is added.

When the Number of Cycles 15000 cycles, Noted NR N-CC0 reach to 6mm, and reduce to 5.5mm at NR-NCC3, and 5.25mm at NR-CC5, and 5mm for both NR-NCC7 and NR-NCC10.

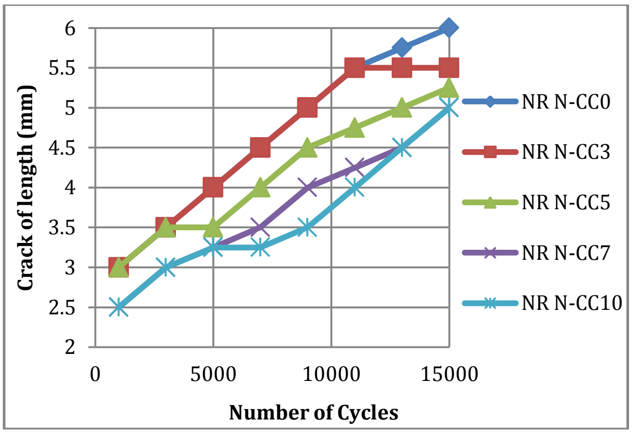

Fig. (3) The crack length against number of cycles for NR compound.

Note in Figure (4) that there is a correspond among of all NR compounds in the region crack initiation I (CI) and stable crack growth II (SCG and the variation in the region unstable crack growth III (UCG).

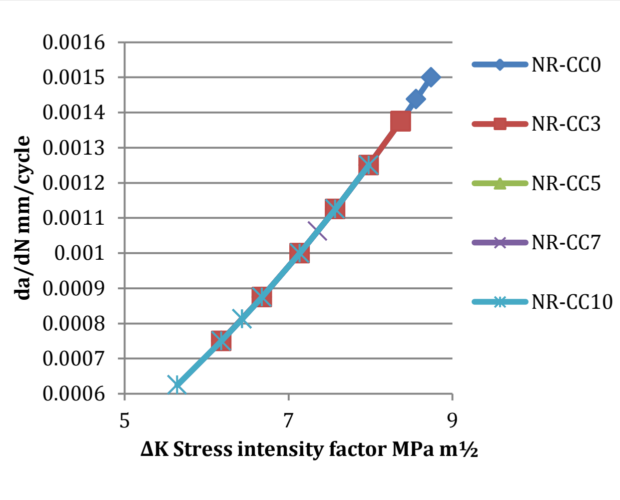

Fig. (4) The crack length against number of cycles for NR compound

Conclusions

1) The tests in which the convergence of particles such as crack growth fatigue test, there is a significant impact and improved properties.
2) The crack growth fatigue test shown that the crack length is decreased with the increasing of percentages of Calcium Carbonate Nanoparticles loading CaCO3.
3) There are better values and approach between the compound of used in this search compared to other compounds with a ratio of high Carbon Black.
4) The there is a convergence as significant between the values of NR/N-CC7 and NR/N-CC10 compounds, particularly at the number of cycles 13000 and 15000 cycles.
5) The minimum value of crack growth fatigue test at NR/N-CC7 and NR/N-CC10 is 4.5 mm at 13000 cycles and 5 mm at 15000 cycles.
6) Influence the Calcium Carbonate CaCO3 nanoparticles (N-CC) beginning at 13000 cycles and up.

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