Chapter 1

Introduction, Literature Review and Objective of the Research Work

1.1 Introduction

Positive temperature coefficient to resistivity (PTCR) is an interesting phenomenon in the field of conducting polymer composites (CPC) consisting of an insulating polymer matrix and conducting fillers. At room temperature the resistivity of the PTCR material is very low. In the case of an over current situation, resistivity of the material rises within the positive temperature coefficient region (PTC trip temperature). This additional resistivity in the circuit has the effect of reducing the overall current. Once the over current situation has been removed, the PTCR material will cool, in doing so its internal temperature drops resulting in the resistance returning to a low state.

The technology of over-current protection in designing a circuit can be accomplished with the use of either a traditional fuse or the more recently developed resettable PTCR polymer composites. Understanding the differences between these two components simplifies choosing the PTCR polymer composites as the best protection device for the application. While, fuses have been referred to as "one time" devices (the fuse provides protection from the overload by opening only once and then it needs to be replaced), the PTCR polymer composites also reacts similarly to the excessive current but is known as a "resettable" device. The polymer based unit can provide over-current circuit protection a number of times when resets by removing the overload.

1.2 Problem Definition and Research Issues

The PTC trip temperature of most PTCR polymer composites is associated with the melting temperature (T_m) of semicrystalline/crystalline matrix polymer and the glass transition temperature (T_g) of the amorphous matrix polymer. Thus, the dimensional stability of the matrix polymer near the transition temperature (T_g or T_m) is a major concern that leads to the disadvantage of PTCR polymer composites. At the PTC trip temperature the continuous conductive network of the fillers breaks and upon cooling, the same network may not form. Thus, upon recycling, the PTCR polymer composites do not show the initial room temperature resistivity. This non reversibility and non repeatability of the PTCR composites limits its applications in high-voltage situation. Most of the reported PTCR polymer composites are also associated with decrease in the resistivity immediately after the PTC trip temperature, known as negative temperature coefficient to resistivity (NTCR) effect. This limits the application of PTCR materials as over-temperature protector and as current limiters. Thus, development of PTCR polymer composites with improved performance properties (repeatability and reversibility, elimination of NTCR effect, dimensional stability etc.) is of great importance which provides the scope for further investigation on PTCR polymer composites.

1.3 Basics of Composites

1.3.1 Composites

Composites are heterogeneous materials created by combining chemically distinct materials in order to improve the properties or to create materials with desired properties. A composites material is made by combining two or more materials to give a unique combination of unusual properties. Composites material is composed of two or more distinct phases (continuous matrix phase and dispersed phase) and having bulk properties significantly different form those of any of the constituents.

Matrix Phase: The primary phase, having a continuous character, is called the matrix. Matrix is usually more ductile and relatively less hard phase. It holds the dispersed phase and shares a load with it.

Dispersed (reinforcing) phase: The second phase (or phases) is embedded in the matrix in a discontinuous form. This secondary phase is called dispersed phase. Dispersed phase is usually stronger than the matrix, therefore it is sometimes called reinforcing phase.

Depending on the matrix phase, composites are broadly classified into 3 types: polymer matrix composites, ceramic matrix composites, and metal matrix composites.

1.3.2 Polymer Composites

Polymer composites are made by incorporating a reinforcement system and (or) fillers to the polymeric material. The resulting composites show properties, both mechanical and functional different from the base polymers used. Polymer composites are very popular due to their low cost and simple fabrication methods. Polymers can also be molded into any complex shapes and are durable materials. Polymers containing conventional fillers (such as carbon black, nanoclay, carbon nanotubes, glass spheres, short fibers, continuous fibers, or talc particles) are known as polymer composites.

1.3.3 Conducting Polymer Composites (CPC)

Electrically conducting polymer composites (CPC) are usually prepared by incorporating conductive fillers such as carbon black (CB), carbon fiber, metal powders, graphite and carbon nanotubes (CNTs), into insulating polymer matrix above certain concentration known as percolation threshold (above which insulating polymer become conducting) [1-3]. The CPC exhibits a series of unique features, such as comparatively low room temperature resistivity, a percolation phenomenon, dependence of resistivity to temperature, pressure and gas, and a nonlinear voltage-current relationship [1, 4-7]. Various CPCs have been developed for numerous applications, such as light emitting diodes, batteries, antistatic coating or fibers, corrosion protection, activators electromagnetic interference shielding, self-regulating low-temperature heater, self-resetting over-current protection elements, electrostatic charge dissipation and many different sensors for vapors, chemicals, mechanical stress and temperature [8-10]. PTCR is the most important and interesting phenomena shown by the CPC, where the resistivity of the CPC increases at certain temperature (PTC trip), making the composites electrically insulating one. The PTCR phenomenon in polymer composites was first observed by Frydman in 1945 [11].

1.3.4 Positive Temperature Coefficient to Resistivity (PTCR)

A sharp rise in resistivity in CPC near the T_m of a semicrystalline or crystalline polymer matrix and T_g of an amorphous polymer matrix is known as PTCR characteristics of the CPC. PTCR materials behavior is characterized by insignificant change in resistivity up to a certain temperature and then a sudden increase in resistivity by several orders of magnitude over a very small temperature range. This temperature is called the PTC trip or switched off temperature. Semiconducting ceramic materials also exhibit PTCR behavior at a certain temperature, known as curie temperature. In CPC, the nature of the filler (shape and size), the structure and crystallizability of the matrix polymer, the interaction between the filler and polymer, and the processing conditions were believed to be the prominent factors relevant to not only the percolation but also the PTCR behavior. A desired PTCR performance includes a low room-temperature resistivity, a high PTC intensity (ratio of maximum resistivity to room temperature resistivity), a low NTC intensity (ratio of maximum resistivity to resistivity at maximum temperature), a high repeatability and reproducibility, and together with a low conductive filler content [12-14]. In PTCR polymer composites, the continuous network structure of filler-filler particles is responsible for the conductivity. Thus, disruption in filler-filler particle contact led to sudden rise in electrical resistivity near the PTC trip temperature where adequate volume expansion (required for particle-particle separation) of the matrix polymer takes place. On the other hand, the NTCR effect is presumably due to the re-aggregation (agglomeration) of the conducting particles in the molten polymer (near T_m) and reformation of the conductive pathways.

1.3.5 PTC Intensity

PTC intensity (I_{PTC}) is defined as the ratio of the maximum resistivity (ρ_{max}) to the resistivity at room temperature (ρ_{RT}) calculated from the temperature dependence of composite resistivity, as shown in Equation 1:

$$\mathrm{I}_{PTC} \approx \frac{\rho_{\max}}{\rho_{RT}} \qquad (1)$$

The PTC trip temperature of the CPC, can be effected by: (1) the natures between conductive filler and polymer matrix; (2) the dispersion quality of conductive filler in composites; (3) the interfacial character in between the conductive filler and matrix; (4) the mismatch in CTE between conductive filler and matrix, and (5) the processing condition and thermal histories of the CPC. Because of their high technological electrical and electronic applications and for commercial interest, polymeric PTCR materials have been studied extensively worldwide since many decades. Polymeric PTCR composites are extensively used in wide range of applications [12-17] in industries and home appliances, such as:

- Over-current protection
- Over-temperature protection
- Portable electronics over-current protection
- Industrial controls over-current protection
- Self-regulating heaters
- Resettable Fuses

1.4 Literature Survey on PTCR Polymer Composites

Since the discovery of PTCR effect in polymers by Frydman in 1945 in low-density polyethylene (LDPE) loaded with CB, many investigators have extensively studied the PTCR effect in polymers [18-25]. Meyer [18, 19] proposed that conductive filler filled polymer composites with the conductive particles separated by a 30 nm thin crystalline polymer film was more conductive than an amorphous polymer film. At a temperature close to the T_m of the matrix polymer, the melting of the crystalline films resulted in an increase of the particle-particle distance, leading to a remarkable increase in the resisitivity of the composites.

Voet [20] illustrated that the PTCR effect of a CB filled polymer was caused by an increase in the average particle or aggregate distance of the dispersed CB. He proposed that the large thermal expansion upon the melting of the crystallites attributed to the strong PTCR effect in a CB filled semicrystalline polymer composite.

Klason et al. [21] reported the PTC trip temperature above T_g for a number of composites consisting of amorphous polymers, such as polystyrene (PS), poly(methyl methacrylate) (PMMA), polyvinyl chloride (PVC) with CB, and near the T_m in crystalline polymers (PE, polypropylene (PP))/CB composites. The NTCR effect in these composites was explained in terms of the formation of new conductive CB networks above the PTC trip.

Narkis et al. [22] described the improvement in reproducibility of PTCR phenomena in PE/CB composites by using a mixture of CB having different (220, 300, 5000 Å) particle sizes. They observed the PTCR phenomena near the melting region of PE and reported the dependences of PTC intensity on the type of CB.

Narkis et al. [23, 24] also eliminated the NTCR effect with improved reproducibility in PE/CB (0.05, 0.1 and 0.5 vol%)composites through peroxide or radiation crosslinking of the PE matrix. The PTC intensity significantly influenced by the degree of crosslinking, while the PTC trip temperature was independent of the extent of crosslinking of the PE.

Narkis et al [25] also studied the PTCR characteristics of crosslinked high-density polyethylene (HDPE) loaded with conductive CB (100-500 Å) and carbon fibers (CF) as a function of filler concentration and reported the thermoelectric switching phenomenon (PTC trip) in the vicinity of the T_m of the HDPE.

Most of the reported works on PTCR polymer composites are based on the CB-filled single semicrystalline polymer matrix due to their potential industrial applications [26-27]. It has been found that uncrosslinked semicrystalline polymer/CB composites exhibit a sharp NTCR effect, which is a great disadvantage for their application in over-temperature protections [28]. To eliminate the NTCR effect, one successful approach is to crosslink the semicrystalline polymer matrix by peroxide, gamma radiation or electron beam [23-25, 29-34, 35-40].

Xi et al. [35] reported enhanced PTC intensity without any NTCR effect in low molecular weight polyethylene (LMWPE)/ultra-high molecular weight polyethylene (UHMWPE)(90/10 W/W)/CF (23.5 wt%) composites compared to the LMWPE/UHMWPE(90/10 W/W)/CB (6.5 wt%) composites. The PTC intensity reached to 10^9 and PTC trip temperature reached to 116 $^{\circ}$C for LMWPE/UHMWPE/CF (23.5 wt%) composites.

Xie et al. [36] investigated the influence of radiation crosslinking on the PTCR effect on the melt-mixed LDPE/CB (16.7 wt%) composites. They reported that the 400kGy dose of ^{60}Co γ-ray radiation in LDPE/CB (16.7 wt%) composites effectively eliminated the NTCR effect and increased the PTC intensity.

Tang et al. [37] reported an increase in PTC intensity, decrease in NTCR effect and improvement of electrical reproducibility in vinyl polymer/CB composites by radiation crosslinking of the vinyl polymer.

Lee et al. [38] studied the effect of ^{60}Co γ-ray and electron beam radiation crosslinking of HDPE powder on the PTCR and NTCR behavior of HDPE/CB(40 wt%) composites. NTCR effect, eliminated by 120 kGy γ -ray radiation dose and 70 kGy EB dose in HDPE/CB (40 wt%) composites. The reproducibility was significantly improved for the radiation crosslinked composites.

Xu et al. [39] showed that chemical crosslinking of the UHMWPE by dicumylperoxide (DCP) in the UHMWPE/CB (13 wt%) composites did not have any significant effect on the PTC intensity and reproducibility of the composites. And the PTC trip temperatures of both (uncrosslinked and crosslinked) UHMWPE/CB (13 wt%) composites appeared at 160 °C higher then the melting point (135 °C) of composites.

Jia et al. [40] investigated the influences of ^{60}Co γ-ray and electron beam dose (1-400 Mrad) irradiation on the PTCR effect of (3:1:1) LDPE/ethylene-propylene-diene terpolymer (EPDM)/CB composites. Crosslinking of the composites by irradiation of 4 Mrad ^{60}Co γ-ray dose eliminated the NTCR effect and improved the PTC intensity by 4 orders of magnitude compared to the uncrosslinked sample, whereas 300 Mrad electron beam irradiated sample showed excellent PTC performance.

Kohler [41] suggested that in a conductive filler filled semicrystalline composites, the conductive filler forms a continuous conductive network structure at a temperature below the T_m of the semicrystalline polymer. On heating, the expansion of the matrix polymer leads to increase the inter-particle distances, resulting in a gradual increase in the resistance of the composites. He explained the PTCR phenomena of the composites near the T_m of the semicrystalline polymer matrix in terms of maximum volume expansion of the matrix polymer which was enough to break the filler-filler continuous network structure.

Ohe et al. [42] studied the PTCR effect of melted mixed paraffin/graphite powder and polyethylene (PE)/graphite powder composites. They suggested that the PTCR effect depends on the inter grain gaps between graphite particles. The uniform distribution of the interparticle distances in the composites changes into a random distribution above the T_m of the polymer. Thus electron tunneling through the inter-grain gaps separating the graphite particles above the T_m becomes difficult, that leads to increase in the resistivity of the composites.

Huali et al. [43] significantly improved the PTC intensity, repeatability and reproducibility of PE/CB composites by crosslinking the PE matrix with ^{60}Co γ radiation. However, the PTC trip temperature of the composites was observed near the T_m of the matrix polymer.

Allak et al. [44] investigated the I-V characteristics of chemically (silane) crosslinked PE/CB (28 wt%) composites at room temperature and above the T_m of PE. The composites displayed a strong PTCR effect near the T_m of PE, with a resistivity jump of four orders of magnitude. They proposed a new model explaining the co-operative effect of changes in crystallinity and volume expansion on the PTCR effect.

Chen et al. [45] investigated the PTCR behavior of LDPE/polypyrrol (PPY) composites, as affected by the loading of PPY (15-40 wt%). They observed the PTC trip of the composites near the T_m of LDPE. However, in the PS/PPY (35 wt%) composites the PTC trip was appeared above the T_g of PS matrix.

Matsushige et al. [46] investigated the PTCR behavior of ethylene-ethyl acrylate copolymer (EEA)/CB (40 wt%) composites and reported that the PTC trip of the composites was appeared at above a critical temperature of about 82 ^{o}C. They suggested that the electron tunneling or hopping processes through the dispersed CB particles was the governing mechanism for the organic PTC compounds rather than the electron transport through the contacted CB particles.

Modine et al. [47] investigated the effect of particle size and CB loading on the room temperature resistivity and PTC intensity of HDPE/CB (18, 50, 82, 101 and 300 nm) composites. For a particular particle size CB, increase in CB loading led to increase in room temperature resistivity. They found the PTC trip near the T_m of the matrix polymer for all the composites. They observed increase in room temperature resistivity and PTC intensity in the HDPE/CB composites with constant CB loading. They also reported that HDPE/CB composites with mixture of different particle size CB showed decrease in room temperature resistivity and PTC intensity compare to composites with particular CB.

Tang et al. [48] studied the effect of the extent of CB dispersion on PTCR characteristics of EEA/CB (25 wt%) composites. The fine dispersion of CB in EEA matrix of the composites prepared by melt-mixing showed strong PTCR effect and high dynamic elastic modulus compared to that of solution-mixing composites having relatively poor dispersion of CB. They proposed that the strong interaction between the polymer and CB is essential for composites to have a high PTC intensity, good electrical reproducibility and high dynamic elastic modulus.

Tang et al. [49] also reported that the PTCR effect in melt-mixed LDPE/CB (20 wt%) composites was related to the largest value of CTE of the matrix polymer near the melting point of LDPE, and the NTCR effect caused by a decrease of the elastic modulus of the matrix polymer. They showed that increase in the elastic modulus or the interaction between CB and LDPE by ^{60}Co-γ-ray radiation crosslinking of LDPE reduced the NTCR effect, and thus improved the PTCR effect and the electrical reproducibility of the composites.

Yang [50] showed significant improvement in PTCR reproducibility, without any NTCR effect, in radiation crosslinked (75/15/10/16) HDPE/EPDM/ethylene vinyl acetate (EVA)/CB composites than that of the un-crosslinked one.

Chan et al. [51] observed PTCR phenomena in UHMWPE/CB (3 wt%) composites near the T_m of UHMWPE, without any NTCR effects. They explained the observed PTCR effect as a result of increase in inter particle distance due to maximum volume expansion near the melting of the crystallites. They also reported that the percolation threshold value and electrical properties of the composites strongly depend on the time and temperature during melt-mixing, viscosity of the matrix polymer, size of the polymer powder and the CB.

Lee et al. [52] explained the PTCR and NTCR effects in annealed HDPE/CB (20 phr) composites on the basis of single parallel resistance-capacitor circuit model by measuring of admittance (current flow) and phase angle. The PTCR effect was characterized by decrease in admittance and increase in phase angle with temperature, whereas NTCR effect was characterized by increase in admittance after the T_m of HDPE. They explained the PTCR phenomenon by the de-agglomeration or the breakage of conduction networks near the T_m region of polymer matrix, and the NTCR phenomenon by the re-agglomeration or the networking of CB aggregates.

Yu et al. [53] reported that the composites of (80/20) LDPE/EVA with CB (15 wt%) showed relatively low room temperature resistivity and high PTC intensity compared to the LDPE/CB (18 wt%) composites. They also found that nitric acid treated CB decreased the percolation threshold and addition of titanate coupling agent increased the percolation threshold of CB in the LDPE/CB composites. Increase in room temperature resistivity and decrease in PTC intensity was observed by nitric acid treatment and titanate coupling TM-931 treatment of CB compare to the untreated one. However, titanate coupling agents (TC-114, TM-27) treated CB decreased both the room temperature resistivity and PTC intensity of the composites.

Yu et al. [54] also reported that treatment of CB with the titanate coupling agent and nitric acid) lowered the percolation threshold of CB and room temperature resistivity of (80/20 w/w) LDPE/EVA/CB (15 wt%) composites. However, irrespective of the CB treatment, the PTC trip temperature was appeared near the T_m of EVA in all the composites.

Feng et al. [55] reported that the room temperature resistivity, PTCR and NTCR effects in PP/UHMWPE/CB (10 wt%) composites were influenced by the weight ratio of the polymers in the composites. When the PP/UHMWPE weight ratio was larger than 3/7, the PTC trip of the composites was appeared near the T_m (≈170 °C) of PP; very similar to the PTCR characteristic of the PP/CB composites. Whereas, the composites with PP/UHMWPE weight ratio equals or smaller than 3/7 showed the PTC trip near the T_m (≈140 °C) of UHMWPE, similar to that of a CB filled neat UHMWPE composite.

Feng et al. [56] also investigated the PTCR effect of (4/1) ethylene tetrafluoroethylene (ETFE)/HDPE/CB (10 wt%) composites. In this composites, the CB filled HDPE particles formed a dispersed phase in the ETFE matrix. A double PTCR effect was observed in the composites, caused by the large thermal expansion due to the consecutive melting of HDPE (near 140 °C) and ETFE (near 215 °C) crystallites.

Yi et al. [57] reported low room temperature resistivity, high PTC intensity, and high PTC trip temperature for annealed PE/CB (35 wt%) composites compare to the virgin composites, suggesting that good thermal-electric switch properties were achieved by the isothermal treatment. Crosslinking by electron beam irradiation of annealed PE/CB (35 wt%) composites at a dose of 20 kGy at room temperature and at 170 °C reduced both the PTC and NTC intensity and decreased the PTC trip temperature than that of unirradiated composites. At energy dose of 200 kGy or 400 kGy the NTCR effect in the composites was completely disappeared. High-dosage irradiation also significantly improved the PTCR reproducibility of the composites.

Dafu et al. [58] proposed a model based on Meyer's theory to explain the PTCR effect of HDPE/CB composites. They reported the PTC trip of HDPE/CB (9.93, 11.9 and 16 vol%) composites near the melting region of HDPE. They explained the phase change as the main cause of the PTCR effect in the crystalline PTCR polymer materials.

Harpaz et al. [59] investigated the PTCR and NTCR effect of poly (4-methyl pentene-1) (TPX)/UHMWPE/CB and TPX/cross-linked XL-UHMWPE/CB composites. Single PTC trip was observed for (70/30 w/w) TPX/UHMWPE/CB (0.67, 1 and 2 phr) composites at about 120 °C. However γ-irradiated UHMWPE in (70/30 w/w) TPX/cross-linked XL-UHMWPE/CB (1.5 and 2 phr) composites showed double-PTC at about 120 and 140 °C. Among all γ-irradiated composites, 220Mrad doses γ-irradiated (70/30 w/w) TPX/UHMWPE/CB (1phr) and (70/30 w/w) TPX/XL-UHMWPE/CB (1.5 phr) exhibited highest PTC intensities.

Kang et al. [60] reported the PTC trip near the T_m of the matrix polymer for HDPE/CB (40 wt%) composites having different particle sizes (23-86 nm), and highest PTC intensity with largest particle size (86 nm) CB in the composites. For a particular CB (23 nm), decrease in CB loading led to increase in PTC intensity of HDPE/CB composites. However, all the composites exhibited the NTCR effect after the PTC trip temperature. They also observed increase in PTC intensity (without NTCR effect) in 150 kGy γ-radiation crosslinked HDPE/CB (40 wt%) composites compare to the uncrosslinked composites.

Luo et al. [61] reported highest PTC intensity in HDPE/CB (27 phr) composites compare to PMMA/CB (38 phr), (25/75 w/w) LDPE/EVA/CB (30 phr) and LDPE/CB (28 phr) composites, and explained it in term of highest crystallinity of HDPE than the other polymers. The PTC trip temperature was related to large expansion accompanying crystalline melting of the matrix polymer. They also reported an increase in the PTC intensity of LDPE/CB (21 phr) composites with increase in annealing time (at constant temperature) and annealing temperature.

Yi et al. [62] investigated the PTCR characteristics of LDPE, LLDPE, PP and polytetrafluroethylene (PTFE) matrix based composites filled with 50 wt% vanadium trioxide (V_2O_3) particle. In all these composites the PTC trip was appeared near the T_m of the matrix polymers, and thus related to maximum volume expansion near the T_m of the corresponding matrix polymer. They also showed that crosslinking could successfully eliminate the NTCR effect in all these composites.

Yi et al. [63] observed the PTC trip near the T_m of respective matrix polymer for PE/CB (0.25, 0.3, 0.4 and 0.5 wt%) and EVA/CB (0.15,0.25,0.3 and 0.35 wt%) composites. They eliminated the NTCR effect of PE/CB and Zinc oxide (ZnO) filled PE/EVA/CB composites by chemical crosslinking using DCP and then radiation crosslinking (^{60}Co-γ-ray).

Feng et al. [64] observed PTCR effect followed by NTCR effect in (50/50 w/w) poly (vinylidene fluoride) (PVDF)/HDPE/CB (7.5, 10 and 20 wt%) composites and the PTC trip temperature ($\approx$140 ^{o}C) in the composites was found to be independent of the CB loading. They explained the PTCR effect by increase in inter-particle distance and reduction in the number of conductive paths due to large thermal expansion of the matrix polymer above the T_m of the HDPE crystallites in the composites. However, with increase in PVDF content in the composites, the room temperature resistivity and PTC intensity were decreased, without any change in the PTC trip temperature.

Lee et al. [65] reported the higher crystallinity of HDPE responsible for higher PTC intensity of HDPE/CB (20, 23, 25, 30, 35 phr) compared to the EEA/CB (18, 20, 25, 30 and 35 phr) composites and the PTC trip temperature of each composites was related to the T_m of the matrix polymer. They also found that (75/25 w/w) HDPE/EEA/CB (14, 15, 17, 20 and 25 phr) composites prepared by melt-mixing of HDPE with EEA/CB had high PTC intensity at the crystalline melting (85 °C) of EEA that that of the composites prepared by melt-mixing (75/25 w/w) HDPE/EEA blend with the CB. Since melt-mixing of HDPE with EEA/CB led to dispersion of CB mostly in the EEA phase, the conduction networks was broken by the melting of EEA prior to the melting of HDPE, and thus PTC trip was appeared neat the T_m of EEA. They also reported reproducibility of composites by annealing the composite.

He et al. [66] reported a low PTC intensity followed by NTCR effect for semicrystalline EPDM/CB (20, 22, 27 30 wt%) composites and only NTCR effect for the amorphous EPDM/CB (22, 26, 30, 32 wt%) composites. They observed the PTC trip temperature of the semicrystalline EPDM/CB composites in the vicinity of T_m of EPDM matrix, irrespective of the loading of CB. By ^{60}Co γ irradiation of both semicrystalline and amorphous EPDM/CB composites, high PTC intensity was appeared without any NTCR effect. The PTC intensity reached as high as six orders of magnitude even for an amorphous EPDM/CB composites and the PTC trip temperature decreased with increasing the irradiation dose.

Yi et al. [67] proposed a phenomenological model to measure the contribution of thermal expansion of polymer to PTC trip temperature of the composites. Using this model, they explained that the PTC trip in HDPE/CB (18.3 and 19.3 vol%) composites was governed by the thermal volume expansion of the composites near the T_m of matrix polymer. The experimental PTCR characteristics of HDPE/CB (18.3 and 19.3 vol%) composites were very similar to the model based theoretical (predicted) data.

Lagreve et al. [68] studied the influence of Poly(buty1ene terephthalate) (PBT) content, CB content and crystallinity of poly(ethy1ene-coalkyl acrylate) (EXA) on the PTCR characteristics of PBT (60~75 wt%)/EXA (X: methyl, ethyl and butyl)/CB (5.55~11.1 wt%) composites. It was observed that the CB was preferentially localized in EXA phase in PBT/EXA/CB composites. Increase in PBT content at constant CB and decrease in CB content at constant PBT in PBT/EEA/CB (5.55~11.1 wt%) composites increased the room temperature resistivity of the composites. In PBT/EXA/CB composites, PTC intensity was found to increase with increasing crystallinity of the conductive EXA phase at low CB content. However, PTC trip temperature in all the above composites was appeared near the T_m of corresponding EXA matrix.

Xi et al. [69] proposed a model to explain the PTCR effect and highest PTC intensity in HDPE/CB composites compare to the LDPE/CB and LLDPE/CB composites. They conclude that formation of larger amorphous region due to the melting of crystalline phase of the matrix polymer was responsible for the PTCR phenomena. They also reported the PTC trip temperature of PVDF/HDPE/CB composites at 120 °C and 165 °C, corresponds to the T_m of HDPE and PVDF, respectively.

Wu et al. [70] reported that the fluorinated CB (FCB) filled PVDF composites exhibited a high PTC intensity and a low NTCR effect compare to the PVDF/CB composites. The PVDF/FCB composites showed a significant PTC intensity at 168 °C, near the T_m of the PVDF matrix. They also showed that increase in fluorine content (above 40 phr) in FCB could eliminate the NTCR effect in PVDF/FCB composites.

Hou et al. [71] investigated the effect of heat-treatment (annealing) on the PTCR effect of (80/20 w/w) LDPE/EVA/CB (18 wt%) composites. They observed an increase in PTC intensity for (80/20 w/w) LDPE/EVA/CB (18 wt%) composites, with increase in annealing temperature (75 ^{o}C to 90 ^{o}C). Annealing of irradiation (by 25 Mrad dose ^{60}Co-γ-ray) crosslinked (80/20 w/w) LDPE/EVA/CB (18 wt%) composites resulted in reduced room temperature resistivity and improved PTC intensity compare to that of uncrosslinked annealed composites.

Park et al. [72] investigated PTCR effect of 30 wt% CB filled polymer composites based on EVA, LDPE, LLDPE, HDPE, and PP matrix. The PTC trip temperatures of all the composites were appeared near the T_m of the corresponding matrix polymer. The largest PTC intensity was observed in HDPE/CB (30 wt%) composites, which was explained with respect to higher volume expansion near the T_m due to higher crystallinity of HDPE.

Zhang et al. [73] reported enhanced PTCR reproducibility of (2/1 w/w) HDPE/EVA/CB (35 wt%) composites because of stronger interaction between polar components, EVA and CB, compared to the HDPE/CB (35 wt%) composites. They also observed improved PTC intensity of (2/1 w/w) HDPE/EVA/oxidized CB (o-CB) (35 wt%) composites than that of (2/1 w/w) HDPE/EVA/CB(35 wt%). This was attributed to the better interaction between EVA and o-CB. However, PTC trip temperature in all the composites was appeared near the T_m of HDPE.

Di et al. [74] studied the effect of mixture of CB and CF on the PTCR and NTCR effect in LDPE/CB/CF composites. They observed enhanced PTC intensity, lower NTCR effect and higher PTC trip temperature for LDPE/(14/7 w/w and 8/8 w/w) CB/CF composites, compared to the LDPE/CB (35 wt%) and LDPE/CF (12 wt%) composites. The NTCR phenomena was effectively eliminated and the PTC intensity was improved by ^{60}Co-γ ray radiation crosslinking of LDPE/(14/7 w/w and 8/8 w/w) CB/CF composites and LDPE/CB (35 wt%) composites with 15 Mrad and 40 Mrad doses, respectively.

Zhao et al. [75] observed the PTC trip temperature of PVDF/CB (8 wt%) composite in the vicinity of the T_m of the matrix polymer. The NTCR effect of the composites was eliminated by crosslinking PVDF with 730 kGy dose ^{60}Co-γ ray irradiation. They reported that during consecutive heating-cooling cycles, slowly cooled PVDF/CB (8 wt%) composites showed higher PTC trip temperature and PTC intensity compared to suddenly cooled PVDF/CB (8 wt%) composites.

Zheng et al. [76] investigated the relationship between the PTCR behavior and the dynamic rheological properties of HDPE/CB composites. The change in resistivity of the composites with increasing temperature was associated with the dynamic modulus of the composites before the PTC and NTC transition temperatures. When the temperature approached to the T_m of HDPE, resistivity of the composites was increased rapidly with a decreasing modulus, corresponding to the PTC transition. The decrease in modulus of the composites during the melting process of the matrix polymer facilitated the relaxation of macromolecular segments and the relative movement of conducting particles.

Park et al. [77] reported the effect of irradiation, thermal ageing and addition of an antioxidant on the PTCR characteristics of HDPE/CB (50 wt%) composites. The PTC intensity of the composites was slightly increased and NTCR effect was eliminated by crosslinking with 100 kGy dose ^{60}Co-γ ray irradiation. The crystallinity of HDPE was changed with thermal ageing (50-140 °C). The lowest room temperature resistivity and the highest PTC intensity was observed for the HDPE/CB (50 wt%) composites aged at 120 °C for 6 h and irradiated at 150 kGy. In radiation crosslinked composites containing (0.5~3 wt%) antioxidant (irganox 1076) showed better thermal stability of the composites. However, increasing amounts of antioxidant led to NTCR effect with decrease in the percentage of crosslinking and the PTC intensity of the composites. Moreover, in all the composites, the PTC trip was observed near the T_m (≈ 133 °C) of HDPE.

Mohanraj et al. [78] reported that the volume resistivity of vulcanized styrene-butadiene rubber (SBR)/CB (10~60 phr) composites was increased with increase in temperature from 40 ^{o}C to 120 ^{o}C and maximum volume resistivity was obtained near the T_m (120 ^{o}C) of SBR. They showed that the PTCR effect in SBR/CB (10~60 phr) composites during the heating and cooling cycle did not follow the same route (phenomena known as electrical hysteresis), which indicated a change in some of the conductive networks during the heating and cooling cycle in the composites. With repeated heating cycles, room temperature resistivity of the composites was decreased, suggesting the formation of more continuous path after heat ageing.

Di et al. [79] investigated the effect of interaction between EVA and CF on the PTCR and NTCR behavior of the EVA/CF (14 wt%) composite. The PTC trip temperature of EVA/CF composites was appeared near the T_m of EVA. A decrease in PTC trip temperature and increase in room temperature resistivity was observed in oxidized CF filled EVA composites due to better interaction of EVA with treated CF compared to that of untreated CF filled EVA composites. The NTCR effect of EVA/CF (14 wt%) composites was effectively eliminated and the PTCR reproducibility was improved in EVA/oxidized CF composites.

Hou et al. [80] reported reduced room temperature resistivity, increased PTC intensity and greatly enhanced PTCR reproducibility of *in situ* melt grafted CB-acrylic acid (30 wt%) filled LDPE composites compared to LDPE/CB (20 wt%) composites. This was due to the stronger interfacial interaction between CB and LDPE through in situ grafting.

Di et al. [81] studied the effects of loading and aspect ratio of CF on the PTCR characteristics and room temperature resistivity of LDPE/CF (9~16 wt%) composites. Increase in CF content reduced the room temperature resistivity, PTC intensity and increased the PTC trip temperature of the composites. The composites containing high content or high aspect ratio CF exhibited a lower room temperature resistivity and PTC intensity. However, the composites containing low CF content or low aspect ratio CF exhibited a higher room temperature resistivity and PTC intensity. A balance between the PTC intensity and the room temperature resistivity was achieved by using a mixture of low and high aspect ratios CFs. The γ-ray radiation crosslinked composites exhibited a higher PTC intensity and PTC transition temperature with out NTCR effect than the uncrosslinked counterpart. However, the PTC trip temperature of uncrosslinked composites was appeared near the T_m of the matrix polymer.

Wan et al. [82] investigated the effect of processing parameters, such as mixing time and temperature, nip gap in two-roll mixer, on the PTCR characteristics of SBR/CB (40~53 wt%) composites. At constant CB loading, conductivity of the composites increased with increase in mixing temperature. The composites prepared with longer mixing time (more than 10 min) exhibited higher resistivity and very poor PTCR property. At 60 °C mixing temperature and 7 min mixing time, resistivity of the composites was significantly increased with reduction in nip gap of the two-roll mixer. The composites prepared through optimized processing parameters (at 60 ^{o}C, 7 min mixing time, and 4 mm nip-gap) exhibited relatively high PTC intensity and well defined PTCR characteristic.

He et al. [83] observed very high PTC intensity (1.1 x 10^4) in HDPE/MWCNT (5.4 wt%) composites. However, the PTC trip temperature of the composites was appeared near the T_m of HDPE matrix, followed by a small NTCR effect above the T_m of HDPE. Crosslinking of the HDPE matrix by 80 kGy ^{60}Co-γ-ray irradiation could eliminate the NTCR effect, without affecting the PTC trip temperature of the composites.

Narkis et al. [84] studied the PTCR characteristics of a thermosetting composites system consisting of epoxy resin (based on di-glycidyl ether of bisphenol A with tri-ethylenetetramine (DGEBA) with tri-ethylenetetramine (TETA) curing agent) and copper coated chopped glass fibers and mica flakes. They reported the percolation threshold in these systems at very low copper contents of 0.11~0.44 vol%. The epoxy/copper coated mica (12 vol%) thermosetting composites exhibited significantly high PTC intensity about 9th order magnitude, without any NTCR at 74 ^{o}C above the T_g (68 ^{o}C) of composites. This was due to the highly crosslinked epoxy matrix that restricted the movement of the filler particles during the heating cycles. The PTC trip temperature of the composites decreased on repeated heating-cooling cycles, indicating that the PTCR property of the composites was not repeatable. They explained the mechanism of the PTCR effect in the epoxy/copper coated mica composite in terms of larger thermal expansion coefficient of the matrix compared with the ceramic filler.

Zhang et al. [85] studied the PTCR characteristics of the UHMWPE/CB composites. They showed that increase in molecular weight of UHMWPE, and decrease in the particle size of CB lowered the percolation threshold of the CB in the composites. For relatively lower molecular weight UHMWPE (145M)/CB (2~5 wt%) composites, the PTC trip temperature was observed at 90 ^{o}C, much lower than the T_m (≈129 ^{o}C) of UHMWPE, and the composites was also characterized by NTCR effect. However, at high CB loading (6 wt%), the PTC trip temperature of the composites was observed near the

T_m of UHMWPE, without any NTCR effect. The higher molecular weight UHMWPE (630M)/CB (0.6~10 wt%) composites was characterized by a double PTCR effect; the first PTCR effect was observed near the T_m of UHMWPE and the second PTCR effect took place at ≈210 ^oC without any NTCR effect. The higher viscosity of higher molecular weight UHMWPE (630M) lowered the degree of intermixing, leading to the formation of stronger conductive paths in the composites relative to the 145M UHMWPE system. Thus, broken up of the conductive pathways near the T_m of the matrix was responsible for the first PTCR effect for higher molecular weight UHMWPE (630M)/CB (0.6~10 wt%) composites. At temperature above 200 ^oC, reduction in viscosity of 630M UHMWPE melt resulted in a higher degree of intermixing and thus, the second PTCR effect was attributed to inter mixing between UHMWPE and CB.

Boiteux et al. [86] studied the PTCR characteristics of PP/co-polyamide (CPA)/iron (Fe) composites prepared by the diluting the CPA-Fe (35 vol%) masterbatch with PP. The PP/CPA-Fe (6~10 vol%) composites having a co-continuous networks of PP and CPA-Fe showed the PTCR characteristics in between those of CPA-Fe (35 vol%) masterbatch and pure PP. The PTCR characteristics of PP/CPA-Fe composites with Fe content more than 15 vol% was similar to that of CPA-Fe (35 vol%) composites. However, all the prepared composites with Fe content above 15 vol% showed the PTC trip at ~80 ^oC, close to the T_m of CPA. This was due to the selective dispersion of Fe particles inside the CPA phase of the composites.

Lee et al. [87] investigated the effects of the addition of MWCNTs on the PTCR characteristics of conventional HDPE/CB (20, 25 and 30 wt%) composites. The HDPE/CB (25 wt%)/MWCNT (0.5 wt%) composites showed highest PTC intensity among the hybrid nanocomposites containing CB (20, 25 and 30 wt%) and MWCNT (0.5~2.5 wt%) composites. They also reported improved PTC intensity and repeatability of the HDPE/CB (25 wt%)/MWCNT (0.5 wt%) composites compared to HDPE/CB (25 wt%) composites. This was due to increase in the degree of crystallinity of HDPE in the presence of MWCNT. However, all the prepared composites showed the PTC trip temperature near the T_m of HDPE.

Xu et al. [88] studied the effect of heat treatment on the PTCR characteristics of polyethylene terephthalate (PET)/CB microfibrillar filled HDPE composites. The isothermal heat treated (3 min at 180 ^{o}C) PET/CB microfibrillar filled HDPE composites showed a strong PTCR effect of 5 orders of magnitude near the T_m (130 °C) of HDPE, compared to the untreated composites. The PTC intensity of the composites reduced with increase in isothermal treatment time. However, there was no effect of isothermal treatment time on the PTC trip temperature of the composites.

Mamunya et al. [89] reported the double PTCR effects in the composites based on HDPE/polyoxymethylene (POM) blends filled with 9 vol% of Fe. The first PTC trip was observed near the T_m (130 ^{o}C) of HDPE, while the second PTC trip (at 154 ^{o}C) was associated with the melting of POM. The microstructure of the composites revealed the presence of Fe selectively in the POM phase and formation of a co-continuous structure of POM-Fe and HDPE. Thus, the double PTCR effect was explained in terms of thermal expansion and melting process, where the first PTC trip was due to the thermal expansion of the nonconductive HDPE phase near its T_m which resulted in a discontinuity of the conductive POM-Fe phase; the second PTC trip originated from the break of the Fe-Fe conductive structure inside the POM-Fe phase in the vicinity of the T_m of POM.

Pourabbas et al. [90] investigated the effect of annealing on the PTCR effect in HDPE/CB (20~35 wt%) composites by using both unmodified CB and the CB modified with metal particles (Au and Ni). The PTC intensity of all the prepared composites was improved after annealing. This was explained considering the improved crystallinity and crystallite sizes of the matrix polymer (HDPE) during the heat treatment that led to a different melting behavior of the HDPE matrix. They also reported very small variation in PTC trip temperature for the entire studied composite after annealing.

Song et al. [91] investigated the effect of annealing at various temperatures from 116° to 149°C (covering the PTC transition during heating) on the PTCR characteristics of the HDPE/CB (0.113 vol%) composites. Annealing the composites resulted in an increase of room temperature resistivity and slightly decreased PTC intensity, compared to that of HDPE/CB composites without annealing. However, the PTC trip temperature of the composites, without and with annealing, was appeared near the T_m of HDPE.

Chang et al. [92] studied the PTCR characteristics of poly(styrene-co-n-butylacrylate)/Nickel plated core-shell poly(styrene-codivinyl benzene) (15~25 vol%) composites. The PTC trip temperature of the composites was appeared before the T_g of the matrix polymer poly(styrene-co-n-butylacrylate). The higher thermal expansion of the polymer matrix even below the T_g of the polymer matrix during heating could disrupt the conductive path of the Ni-plated core-shell poly(styrene-codivinyl benzene) particles.

Zhou et al. [93] studied the PTCR effect in LLDPE/poly(ethylene-methyl arylate) (EMA)/CB composites. They reported that the CB was located preferentially in the nonpolar LLDPE phase due to the lower viscosity of LLDPE compared to EMA, although the interaction or interfacial energy between CB and EMA is stronger than that between CB and LLDPE. The PTC trip temperatures of (80/20 and 20/80 w/w) (LLDPE/EMA)/CB (30 wt%) composites were appeared near the T_m of the major phases.

Shen et al. [94] proposed a mathematical model to compare the PTCR effect of HDPE/CB (0.131 and 0.149 volume fraction) and PS/CB (0.258 and 0.277 volume fraction). It was found that the model was more applicable for PS/CB composites than HDPE/CB composites. They found the PTC trip near the T_m of HDPE for HDPE/CB composites and near the T_g of PS for PS/CB composites. Due to higher volume expansion of HDPE near the T_m compared to that of PS near T_g, the HDPE/CB composites showed higher PTC intensity than that of PS/CB composites. They explained the PTCR effect of all the prepared composites in term of thermal volume expansion of the matrix polymer.

Kalappa et al. [95] reported better PTCR effect of polyaniline (PANI) functionalized MWCNT (0.5, 1 wt%) filled HDPE/CB (20, 25 and 30 wt%) composites compared to unfunctionalized MWCNT filled HDPE/CB(20, 25 and 30 wt%) composites. They showed that the PTC trip of all the functionalized and nonfunctionalized composites was appeared near the T_m of HDPE matrix. This was due to the significant volume expansion in the composites near the T_m of the HDPE in presence of hybrid fillers and a sudden increase of the resistivity was due to the disconnection of the conductive paths. However, the maximum PTC intensity was observed for the composites formulated with 25 wt% CB and functionalized MWCNT (0.5 and 1 wt%).

Zhang et al. [96] reported double PTCR effect in HDPE/(60/40 w/w) Sn-Pb (35 and 40 wt%) composites without any NTCR effect. The Sn-Pb alloy was treated with 10 wt% titanate solution. The first PTC transition in the composites was related to the T_m of the HDPE matrix, whereas the second PTC transition was appeared at a temperature (183 ^oC) close to the T_m of the Sn-Pb alloy. Increased PTC transition temperature, decreased the PTC intensity and improved the reversibility and reproducibility of the double-PTC behavior of HDPE/Sn-Pb composites was observed after surface treatment of Sn-Pb alloy with titanate concentration ≤1wt%. However, lower PTC transition temperature, higher PTC intensity and poor reversibility and reproducibility of the composites was observed by the surface treatment of Sn-Pb alloy with more than 1 wt% titanate concentration.

Su et al. [97] investigated the effects of modification of CB on the on room temperature resistivity, PTC intensity, and PTCR characteristics of the composites based on LDPE and PANI modified CB (CB-PANI). They showed that CB modified with PANI improved the compatibility and dispersion of CB particles in the LDPE matrix, which resulted in a decrease of the percolation threshold and improvement of reproducibility of the PTCR effect in the composites. The PTC trip temperature of the composites was found near the T_m of the LDPE.

Chen et al. [98] studied the PTCR properties of LMWPE/UHMWPE/CF (23.5 vol%) composites prepared by the gelation from solution and kneading in the melting state. The (9/1 w/w) (LMWPE/UHMWPE)/CF (23.5 vol%) composites showed the PTC trip at 116 ^oC with highest PTC intensity and lowest room temperature resistivity, compared to the composites. For the composites with (3/1 and 6/1 w/w) LMWPE/UHMWPE, CFs were selectively localized in the mixed region of LMWPE and UHMWPE, whereas in the (9/1, 12/1, and 15/1 w/w) LMWPE/UHMWPE composites

CFs were dispersed mainly in the region of LMWPE. Thus, the highest content of CF within LMWPE region in the 9/1 composite and the 9/1 composite provided the most significant PTCR effect due to the large thermal expansion of LMWPE. The high melt viscosity of UHMWPE inhibits the movement of CFs and eliminated the NTCR effect in the composites.

Zhang et al. [99] prepared the conducting multiphase polymer composites of (76/19/5 w/w) LDPE/PP/CB by melt processing technique. The ternary composites exhibited a lower percolation threshold (5 wt%) and a strong PTC intensity compare to binary composites of PP/CB (7.5 wt%) and LDPE/CB (7.5 wt%). The PTC trip temperature of the ternary composites was appeared below the T_m of LDPE, while LDPE/CB and PP/CB binary composites showed the transition near the T_m of the matrix polymers.

Bao et al. [100] investigated the PTCR effect of melt-mixed PP/MWCNT (0.5 and 1 wt%)/montmorillonite (MMT) (1~9 wt%) composites. They observed an increase in the PTC trip temperature of the PP/MWCNT (1 wt%)/MMT composites from 110 °C to 152 °C with increasing MMT loading (1~9 wt%). The increase in the PTC trip temperature of the composites in the presence of clay has been explained considering the reduction in thermal expansion of the matrix polymer in the nanocomposites, which required relatively higher temperature to break the continuous conductive network in the PP matrix.

Gao et al. [101] investigated the effect of MWCNT loading, processing temperature, and the annealing time on the PTCR and NTCR effect of UHMWPE/MWCNT (0.2, 1, and 2 wt%) composites. The PTC intensity of the composites was decreased with increasing the MWCNT content and PTC trip temperature was increased from 136.9 ^{o}C to 143.7 ^{o}C with increasing the MWCNT concentration from 0.2 wt% to 2 wt%. They showed that higher molding temperature could lower the viscosity of UHMWPE and thus, formed more compact and perfect conductive network of MWCNTs. Increase in molding temperature from 160 ^{o}C to 200 ^{o}C was found to reduce the PTC intensity of the composites. Whereas, the PTC intensity of the composites molded at 160 °C was increased with increasing the annealing time. However, processing temperature and the annealing time had no significant effect on the PTC trip temperature of the UHMWPE/MWCNT (0.2, 1, and 2 wt%) composites.

Liu et al. [102] reported PTC trip temperature of melt compounded HDPE/bundle-like MWCNT (3, 6 and 9 wt%) composites near the T_m (~140 °C) of HDPE matrix. No NTCR effect was observed for the HDPE/bundle-like MWCNT (3 wt%) composites. However, the NTC intensity increased from 1.18 to 2.45 with increase in MWCNT concentration from 6 to 9 wt%.

Chen et al. [103] studied the PTCR characteristics of melt mixed PP/CB (5 wt%) composites. The PTC trip temperature of the composites was appeared near the T_m of the PP matrix, followed by the NTCR effect. The composites showed unrepeatable PTCR behavior upon thermal cycling. They also reported the PTC and NTC intensities of 6.1 and 6.3, respectively, for the composites.

Li et al. [104] studied the effect of graphite nanofibers (GNF) content on the PTCR behavior of HDPE/CB (15, 20 and 25 wt%)/GNF composites. Significant improvements in the reproducibility and PTC intensity were observed in the HDPE/CB (20 wt%)/GNF (0.25, 0.5, 0.75 and 1 wt%) composites compared to the HDPE/CB (20 wt%) composites. This was attributed to the incorporation of GNFs, minimizing the migration of conducting particles and the shape deformation of the composites. The PTC trip temperature for HDPE/CB (20 wt%) composites and HDPE/CB(20 wt%)/GNF (0.25, 0.5, 0.75 and 1 wt%) composites were observed near 151 °C and 159 °C, respectively slightly higher than the T_m of HDPE. The (80/20) HDPE/CB/GNF (0.25 wt%) composites showed a maximum resistivity (9.578 Ω cm) along with a highest PTC intensity.

Chen et al. [105] investigated the effect of crosslinking and heat treatment on the PTCR effect of the (1/0.25, and 1/1 w/w) UHMWPE/CF composites. The UHMWPE-CF composites containing DCP were prepared by gelation/crystallization, and the resultant dry gel composite films were crosslinked by annealing at 135°C and 220°C. A high

PTCR effect near the T_m of UHMWPE with PTC intensity 10^7 was obtained by heat treatment of (1/1 w/w) UHMWPE/CF composites at 135°C.

Yang et al. [106] investigated the PTCR characteristics of HDPE/EVA/CB (18 wt%) composites with different (0, 20, 60 and 100 wt%) amounts of EVA. They reported that the PTC trip temperatures in all the composites were associated with the T_m of the major phase of the HDPE/EVA blend in which most of the fillers were distributed.

Jiang et al. [107] reported higher PTC intensity and PTCR reproducibility of PVDF/MWCNT (2.5 vol%) composites without any NTCR effect compared to the LDPE/MWCNT (1.7 vol%). They reported the PTC trip temperature at the T_m of PVDF matrix for PVDF/MWCNT (2.5 vol%) composites.

Seo et al. [108] investigated the influence of electro-beam (EB) irradiation on PTCR properties of HDPE/CB (1~5 wt%) composites in a dosage of 30~150 kGy. The EB irradiated HDPE/CB composites showed higher PTC intensity, and good PTCR reproducibility compared to the unirradiated HDPE/CB (1~5 wt%) composites. The PTCR reproducibility of the composites was due to the reduction of free movement of CB particles at above the T_m of HDPE polymer as a result of crosslinking of HDPE in the HDPE/CB composite.

Tsao et al. [109] investigated the PTCR effect of HDPE/CB (25, 33.7 and 37.5 wt%) composites containing 28 wt% aluminum hydroxide ($Al(OH)_3$) as flame retardant. The HDPE/CB/$Al(OH)_3$ composites showed a decrease in room temperature resistivity and increase in PTC trip temperature with increasing the CB content. These composites were also associated with NTCR effect above the T_m of HDPE. The composites with 33.7 wt% CB showed the highest PTC intensity. Increase in the surface activity and the structural completeness of HDPE by argon (Ar) plasma treatment (40 W) enhanced the PTC intensity, without much effect on the PTC trip temperature of the composites. Maximum PTC intensity without any NTCR effect and significantly reproducible PTCR characteristics of the Ar plasma treated (3 min) HDPE/CB (33.7 wt%)/$Al(OH)_3$ (28 wt%) composites was achieved by crosslinking of HDPE with 2 phr DCP and then with 20

Mrad ^{60}Co γ-ray radiation dose. The PTC trip temperatures of all the prepared composites were obtained below the T_m of HDPE.

Costa et al. [110] investigated the PTCR effect of the ethylenebutylacrylate (EBA)/CB (12, 15, 20 and 22 wt%) composites. They observed significant PTCR effect in EBA/CB (15 wt%) composites near the T_g (60 ^oC) of EBA, followed by the NTCR effect. They explained the PTC mechanism by electron tunneling phenomena, where the conductive filler particles were not in physical contact, and the electrons tunnel through the insulating gap between them. They also reported that with repeating heating cycles, the EBA/CB (12 wt%) composites did not show repeatable PTCR effect due to the ageing of EBA matrix.

In summary, most of the previous work on PTCR polymer composites reported that PTC trip temperature is a function of T_g and T_m of the matrix polymer in the composites. Thus, the loss in dimensional stability of the matrix polymer near the transition temperature is a major concern that leads to the disadvantage of PTCR polymer composites. Moreover, the non-repeatable and irreversible PTCR characteristics, the NTCR effect immediately after the PTC trip temperature in PTCR polymer composites are also great problem that limits its application as over temperature and over current protector. This provides the scope for further investigation on developing PTCR polymer composites.

1.5 Objective of the Research Work

In this investigation, efforts have been made to prepare highly reversible and repeatable PTCR polymer composites that can overcome all the aforesaid problems associated with PTCR polymer composites. To explore this, composites were formulated on the basis of the difference in coefficient of thermal expansion (CTE) between the matrix polymer and filler. Significant mismatch in CTE between the polymer and filler at higher temperature, well below the transition temperature (T_g or T_m) of the matrix polymer, may lead to the separation of the conducting particles in the continuous network structure and thus the composites become insulating in nature (PTC trip) without any

NTCR effect and loss in dimensional stability. Thus, the objective of this research is to develop:

- PTCR polymer composites with PTC trip temperature well below the T_m or T_g of the matrix polymer
- Highly repeatable and reversible PTCR polymer composites with improved stability against thermal degradation
- PTCR polymer composites without any NTCR effect
- PTCR polymer composites with high dimensional stability at the PTC trip temperature

1.6 Structure of the Thesis

The thesis is organized into 8 chapters to report the whole research work with the objectives as outlined above.

Chapter 1 of the thesis gives general introduction, problem definition and research issues, basics of composites, polymer composites, conducting polymer composites, PTCR properties in polymer composites, and a comprehensive literature survey. Finally, the objective of the present investigation has been mentioned.

Chapter 2 illustrates the materials details, experimental procedures for the preparation of different types of PTCR polymer composites and various characterization methods for the analysis of different PTCR polymer composites.

Chapter 3 deals with the PTCR characteristics of HDPE/Cu powder composites.

Chapter 4 focuses on the PTCR characteristics of PMMA/Ag-coated glass bead composites**.**

Chapter 5 deals with the PTCR characteristics of PC/Ni-coated graphite composites.

Chapter 6 deals with the PTCR characteristics of SAN/SS composites.

Chapter 7 reports the PTCR Characteristics of PS/Ni-powder/MWCNT Composites.

Chapter 8 gives the summary of the results and conclusion from the investigation.

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