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Review of the Performance Improvement of Electroactive Shape Memory Polymer Composites

Jingyi Zhang*

College of Chemical Engineering, Beijing University of Chemical Technology, Beijing, China

*Corresponding author: 2022010501@buct.edu.cn

Abstract. As an intelligent shape memory material, shape memory polymer (SMP) can achieve long-term memory of temporary shapes and effective recovery of original shapes by means of external conditions such as heat, light and electricity. Therefore, aerospace, biomedical, electronic equipment and other industries not only widely used this technology but also achieved remarkable results. However, SMP has electrical insulation and thermal insulation, which makes the further improvement of its mechanical strength and shape memory performance encounter bottlenecks. Based on this, researchers have made composite materials (SMPC) by adding fillers to shape memory polymers, which not only improves the original electrical and thermal deficiencies of the material, but also improve strength and shape recovery ability, breaking through the development obstacles. However, the problems of temperature limitation and high temperature performance still exist, and thermally active SMPC still faces difficulties in practical use. Based on the above situation, this paper will actively introduce electroactive SMPC, and through the relevant mechanism of shape memory behavior, combined with the modification of electroactive SMPC in detail, summarize the expected research development and future application of electroactive SMPC.

Keywords: Electroactive *SMPC*; SMPC; shape memory behavior; SMP; nano-filler.

1. Introduction

As a kind of smart materials, shape memory polymers (SMP) can remember temporary shapes under the stimulation of heat, light or electricity, and then return to the original state. Based on the characteristics of this material, SMP has great application potential in sensors, packaging materials, smart devices and other aspects [1]. In the field of shape memory materials, shape memory polymer (SMP) is more popular than shape memory alloy (SMA). It is not only more affordable but also has stronger deformability under small load. However, compared with other types of shape memory materials including SMA, SMP has electrical insulation and thermal insulation, and the stiffness and durability of the material itself are low, which makes it can only be applied with low restoring force [2]. Combining the above characteristics of SMP, their material defects limit their use in shape memory applications.

In order to overcome such defects of SMP, people try to introduce various fillers to prepare shape memory polymer composites (SMPC). This effectively improves the strength indexes such as flexibility and torque of SMP, and the prepared SMPC significantly enhances the shape recovery ability and even expands the electrification performance of the material through the introduction of conductive fillers. At the same time, the study found that the characteristics of SMP are not only affected by the contact surface between filler and polymer, but also the processing method will change the performance. Various factors have led to more and more attention to SMPC in recent years [2]. Based on the above factors, people expect the excellent shape memory performance brought by SMPC and are keen to study or discuss thermally active SMPC. It is undeniable that such materials have shown great potential. However, people are not satisfied with the shape recovery of thermoactivated SMPC under high temperature conditions, and the remote-control effect of this kind of SMPC is not good [3]. This feature has prompted many fields such as sensing, tissue regeneration and aerospace to develop electroactive SMPC in depth research in the field: when the voltage triggers the current of the material, the Joule heating effect causes the temperature to rise to achieve shape recovery, and the heat-induced shape memory effect also establishes an indirect heating mechanism [2]. At the same time, the electroactive SMPC is affected by both electrical conductivity and thermal conductivity. In summary, compared with the thermally induced SMPC, the electrically induced SMPC has obvious advantages in remote drive, simple operation and fast response [4]. For example, Claudio Russo et al. [5] compared the shape memory effect of temperature-responsive thiol-acrylate-epoxy SMPC with the electric field-responsive SMPC prepared by the same polymer. The results show that the electroactive SMPC has a faster shape recovery rate than the thermally active SMPC. Among them, 15wt % boron nitride (BN) SMPC can achieve the best recovery effect of 100 % in only 8 seconds. In addition, the recovery of SMPC can be completely controlled by 5 V voltage, and the operating conditions and time cost are relatively friendly compared with the thermally active SMPC.

In summary, based on an overview of the shape memory effect and typical electroactive SMPC research cases, this paper will explore creative material engineering improvement strategies for electroactive SMPC performance, including methods to change surface properties and new fillers to expand the application range of materials [2]. On this basis, this paper will briefly describe the application direction of electroactive SMPC and give a summary and future prospect in this field based on the existing research results.

2. Overview of Shape Memory Behavior

In order to achieve shape memory behavior, it is necessary to control it by programming. Material design must follow the shape memory recovery mechanism and related theoretical laws, so that the original appearance can be restored after deformation. According to the effects of programming and recovery, a variety of shape memory effects have been defined to distinguish different types of shape memory materials. In summary, this section will outline the various programming methods, recovery mechanisms, and main features of different effects of shape memory behavior.

2.1. Programming

In order to obtain the required shape memory characteristics in SMP, the programming process is crucial. After being programmed, SMP can realize the expected deformation actuation, shape recovery and shape shaping after deformation by responding to the stimulation of the surrounding environment or the stimulation of external heat, light, electricity and other conditions. In order to distinguish the types of programmed SMP, according to the material characteristics of SMP, the variation of the loss factor of SMP with temperature is summarized by dynamic mechanical analysis, and the glass transition temperature (T_g) of thermally active SMP prepared by different materials is determined [2]. According to the difference of T_g, programming is divided into three types: (1) cold programming, (2) warm programming and (3) hot programming. This section will briefly describe these three programming methods.

The reason why cold programming has attracted attention is not only because it has higher recovery stress and faster recovery rate than hot programming, but also because the prerequisite for this process is that the SMP deformation must exceed the special requirements of yield strength. Although thermosetting SMP is difficult to achieve cold stretching programming, thermoplastic SMP can relatively easily complete this process in the glassy state. However, cold compression programming is feasible for both types of materials, and its compression mode is more widely applicable. [6].

The thermal programming process includes two steps: temporary shape setting and permanent shape reduction. The material is heated above the transition temperature T_{trans}, resulting in an increase in entropy. On this basis, the deformation ability of the material is improved, and the energy barrier is reduced, so that it is easier to control the material to get the desired shape. At the same time, the external force causes the structure of the material to deform and then begin to cool, maintaining the force and temporary shape stability. This operation can be repeated, so that the material gets a different temporary state, and the material will return to its original appearance. [7].

Temperature programming is an important way to control the glass transition region, and its setting temperature and execution steps will significantly change the parameters or conditions of many processes, such as the printing parameters in fused deposition modeling (FDM) 3D printing [6, 8]. This method is most used in thermosetting plastics due to its low deformability and high brittleness compared to thermoplastics, and at T_g.

2.2. Recovery mechanism

The process of polymer molecular chain shrinking from metamorphosis to ordered memory morphology allows SMP to restore its original shape. This elastic phenomenon, namely entropy elasticity, originates from the simple structure formed during material preparation. The shape of its memory actually corresponds to the most stable state of the material [2]. In this regard, the thermoviscoelastic theory and the phase transition mechanism jointly explain the shape memory effect and the recovery principle, mainly including the molecular chain motion is affected by time, and the phase transition controls the energy storage and release.

The polymer molecular chains are regarded as overlapping slender springs in the thermoviscoelastic theory. This random structure maintains a stable state of entropy. As the temperature increases and exceeds the room temperature, the molecular chain mobility increases and exhibits viscoelastic characteristics. The applied load causes the ' spring ' to compress or stretch, and its force direction is the same as the load, resulting in a decrease in entropy. In the subsequent operation of programming, keeping the spring constant load and reducing the temperature at the same time, the thermal viscoelasticity just obtained by the spring is lost, that is, the polymer molecular chain loses fluidity. In the above process, the storage stress of the stretched chain and the compressed chain becomes the elastic potential energy, and the polymer recovery is restored by the thermoviscoelastic behavior, and the shape memory phenomenon is reasonably explained [9].

The polymer is divided into two parts: the active phase and the frozen phase through the phase transition theory. The active phase is composed of movable bonds and dynamic chains and has deformability. The frozen phase is formed by chemical or physical crosslinking and then the structure remains stable. Therefore, when the polymer is in a glassy state, the frozen phase exhibits a stable molecular chain structure and resistance to further structural changes. When the polymer changes from glass-like to rubber-like, part of the frozen phase is transformed into the active phase, and any structural changes of the polymer are stored in the active phase. Subsequently, when the temperature decreases, the local stress of the polymer changes, and the frozen phase is restored. Under low temperature conditions, it is difficult for the polymer to recover shape due to insufficient stress. After the temperature rises, the process of turning the frozen phase into the active phase begins again, and the stored stress is released to make the material change back to its original state [10]. From the thermodynamic point of view, the phase transition between the frozen phase and the active phase is the phenomenon of glass transition. This phase transition process clearly shows how stress storage and release promote shape memory behavior.

2.3. Shape Memory Effect

SMP shape memory cycle includes programming operation and recovery mechanism, which makes SMP often appear two states of initial shape and temporary shape in the complete cycle. There are two possibilities for a single temporary shape generated by SMP during programming. Irreversible deformation corresponds to one-way shape memory effect, and reversible deformation corresponds to two-way shape memory effect. Some SMPs with adaptive molecular structure can display more than two forms after multiple programming and recovery operations. This phenomenon is called three-way or multi-way shape memory effect. For example, bidirectional SMPC belongs to this kind of situation. The specific three shape memory effects are shown in Fig.1 [2].

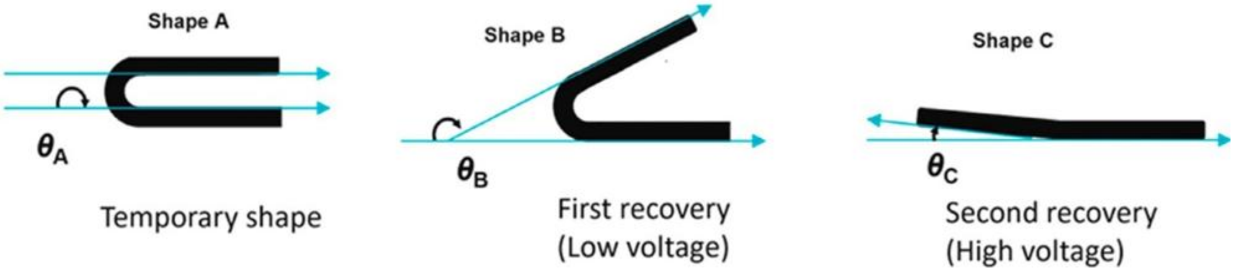

Figure.1. Diagram of bending process of triple shape memory composites [2].

3. Summarization of SMPC Modification Brought by Material Engineering

At present, the research on the modification of SMP is still limited by the mechanical strength and shape memory performance caused by its thermal and electrical insulation. At the same time, people are keen to introduce modified fillers into SMP to prepare thermally active SMPC under easier thermal stimulation conditions to achieve excellent shape recovery performance of SMPC. However, thermally active SMPC will be limited by the high temperature requirements of shape recovery, and even remote-control performance. Therefore, people 's research direction is developing towards the newly manufactured electroactive SMPC, and some teams have studied the influence of changing the proportion of new material components. However, there is relatively little attention paid to the innovative enhancement strategy of the shape memory effect of electroactive SMPC [2]. At the same time, the addition of traditional thermally active fillers has relatively more modification of SMP. Compared with the impact of electroactive fillers, the research of traditional thermally active fillers is still relatively mature. Therefore, in order to achieve higher material properties, people prefer the more potential impact of electroactive fillers on SMPC. Among the electroactive fillers, electroactive nanofillers are favored by people because of their natural nanostructure advantages and conductive nanomaterials for the improvement of shape recovery performance and the expansion of electrification performance of SMPC. Therefore, based on the problems encountered in the process of performance enhancement of SMPC, this section will focus on the effect of nano-filler addition, and on the basis that the shape recovery performance of SMPC has benefited from the introduction of conductive nanomaterials, there is still SMP due to the addition of nanomaterials. The more functional expansion of the material is given, until the introduction of plasticizers has a significant effect on reducing the T_g of SMPC.

3.1. The Addition of Nano-filler

Nanofillers can provide more possibilities for the performance enhancement of SMPC due to their excellent structural characteristics, including increasing the possibility of reducing defect count, surface volume ratio, and enhancing the optical, mechanical and thermal properties of composites [11].

It is well known that most polymers have electrical insulation, including SMP. At the same time, the heat-sensitive SMP is limited by the high temperature requirements of shape recovery and the lack of control effect of shape memory effect, and other forms of signal stimulation are needed to achieve the desired performance [3]. Therefore, graphene, carbon fiber or other nano-fillers with certain electroactivity are actively introduced into SMP. On this basis, the prepared SMPC has shown further improvement in shape memory performance compared with the original SMP due to the addition of electrical signal stimulation. For example, Qi [12] et al. developed a new type of fabric reinforcing material by molding ordinary composite fabric process. The continuous carbon fiber and shape memory polyurethane in the material are used to make core-shell yarn, and the prepared material is referred to as CCF / SMPU. The bending load of this material becomes larger, and the storage modulus is also effectively improved, which is due to the addition of continuous carbon fiber (CCF), which makes its shape recovery rate reach 99.3%. The test also found that the electrothermal recovery performance of the material is good and the deformation ability is stronger.

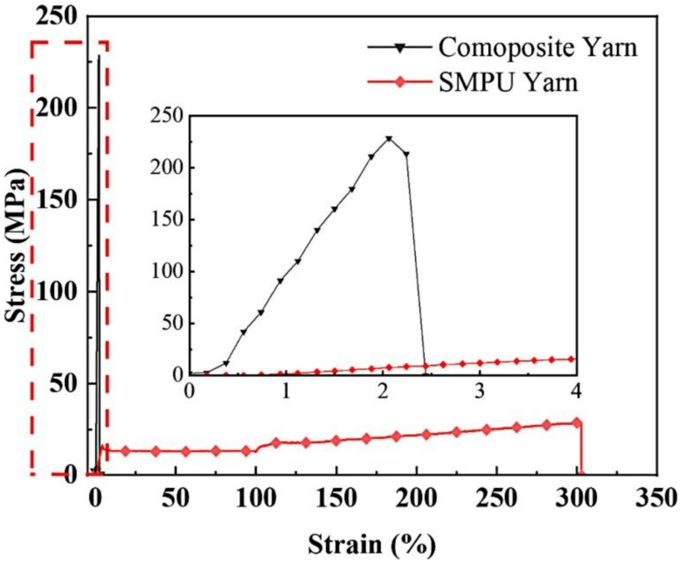

Figure 2. Stress-strain curves of CCF / SMPU yarns and SMPU yarns [12].

As shown in Fig.2, the image shows that the maximum stress of CCF/SMPU yarn is 7.4 times that of SMPU yarn, and brittle fracture occurs after reaching the peak value. The yield behavior of SMPU yarn occurs before the elongation is about 300%, and the tensile strength of carbon fiber reinforced yarn is verified by experiments. In addition, the continuous carbon fiber inside the SMPC forms an effective conductive network, and its good thermal conductivity helps to spread the heat generated by the Joule effect to the entire material. Figure 3 shows the shape recovery process and the corresponding infrared thermal image of the double-layer SMPC under 6V voltage. As the temperature increases, the sample gradually returns to its original shape, and the temperature distribution is uniform during this process. Therefore, SMPC can generate enough Joule heat to reach T_g and show good shape recovery behavior at lower voltage.

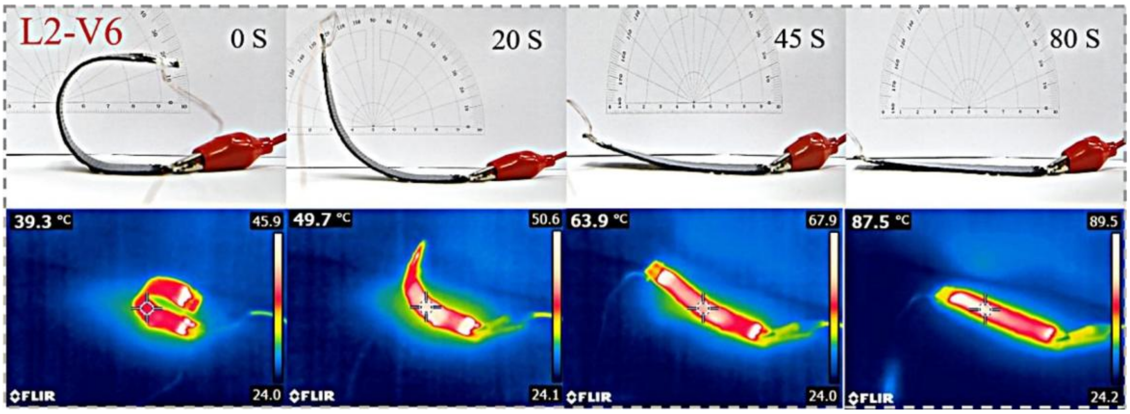

Figure 3. Temperature distribution of two-layer SMPC with CF content of 10.9 % at 6 V during CCF / SMPU recovery [12].

In summary, the addition of nanofillers makes SMPC have higher performance. The very critical mechanical properties of the material and the material recovery performance in the shape memory direction are significantly improved due to the addition of CCF compared with traditional SMPU. As described in this study, the combination of yarn and fabric structure with nano-conductive fillers has good potential in intelligent structural design.

3.2. Functional Expansion of Nanofillers

When improving the performance of SMPC, the poor dispersion of nanofillers has become a common problem, which affects the stable preparation of materials. The research team has improved the dispersion through functionalization. This method has become the key to breaking through the bottleneck. The material system not only optimizes the shape memory performance but also expands the electrochemical performance. Although the combination of nano-filler and matrix has obvious advantages, the dispersion is not enough to lead to unstable performance. Functionalization treatment not only improves the distribution of fillers but also brings new characteristics. For example, surface modification enhances the interfacial bonding force and conductivity, which provides new ideas for the development of multifunctional composites.

The most typical example is that carbon nanotubes (CNT) have been used to enhance electroactivity and functional expansion in the field of electrochemistry. Compared with other fillers, the length-diameter ratio of carbon nanotubes has obvious advantages, and it is easier to form a conductive path, which makes the conductivity of the composite material better. For example, Xu et al. [13] realized the preparation of SMP on the basis of MgMA (Magnesium Maleate) and MAH (Maleic Anhydride) grafted on EPDM/PP (ethylene propylene diene monomer/polypropylene) blends, and compounded CNT after melting of EPDM/PP. CNT has good electrical conductivity and can be uniformly dispersed in EPDM/PP TPV materials due to its small size. Therefore, the prepared Mg-MA or MAH EPDM/PP TPV material is uniformly dispersed by the reaction of the hydroxyl group of carbon nanotubes with the matrix anhydride. Fig.4 shows that the 2Hz conductivity data were tested at 10Hz frequency with the increase of CNT content. This is because the formation of conductive network enhances the overall conductivity of the material. As the content of carbon nanotubes increases, a continuous conductive path is constructed inside the TPV and then the charge transfer efficiency is improved. The distance between the adjacent CNT in the TPV with lower CNT content is larger, resulting in a smaller amount of electron transition between the two CNT, so the conductivity of the TPV is not significantly improved. When the CNT distance is small or the CNT is connected to each other, the electrons can pass freely in the polymer matrix, thereby increasing the conductivity of the TPV. The conductivity of TPV M5C8 reached 1.6×10^{-4}S·cm^{-1}, which was 108 times higher than TPV M5C0.5 (6.2×10^{-13}S·cm^{-1}), indicating that a conductive network was formed in the TPV matrix. The conductivity of TPV M5C2 was 1.9×10^{-12}S·cm^{-1}, which was not only higher than that of TPV M0C2 (7.3×10^{-13}S·cm^{-1}), but also increased by nearly 3 times. The main reason was that the hydroxyl groups on the surface of carbon nanotubes reacted with the anhydride groups of EPDM chains and PP linked branches. The dispersion of carbon nanotubes in the matrix is more uniform to construct an effective conductive network. For example, the SEM image of Fig.5 shows that the dispersion uniformity of carbon nanotubes in TPV M5C2 is significantly better than that of TPV M0C2.

In summary, the introduction of nanofillers into SMP and SMPC achieves the coexistence of shape memory behavior and good electrical conductivity or other properties of SMPC by adding nanomaterials with specific functions (such as CNT, CCF, etc.), expands the application range of SMPC platform and realizes the functional diversity of SMPC material platform. At the same time, the functional combination of CNT and anhydride realizes the uniform dispersion of nanomaterials in SMPC, which solves a major problem restricting its development and application in this field.

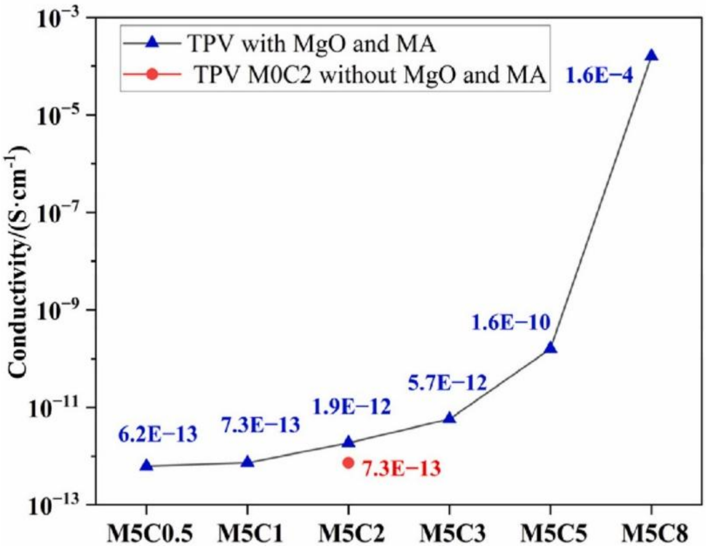

Figure.4. Difference of conductivity of TPV SMPC with different CNT content [13].

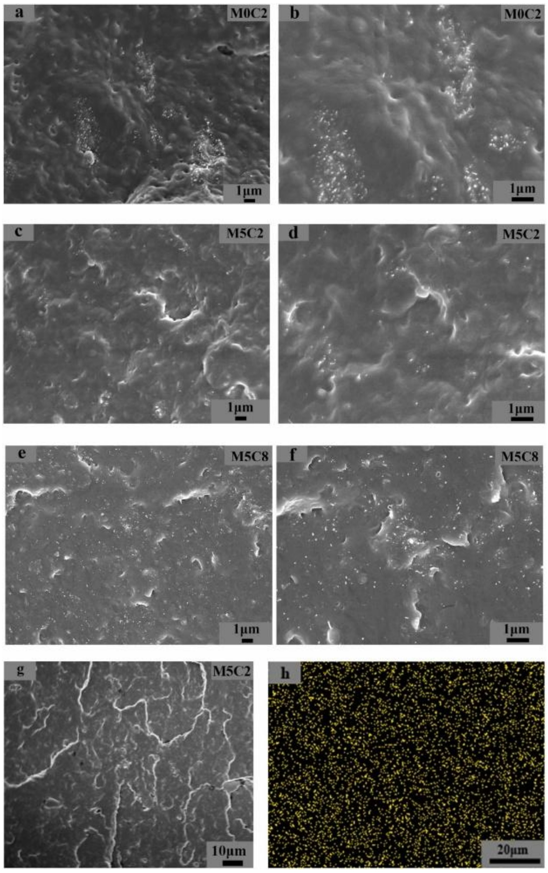

Figure.5. (a–g) The cryo-fractured surface SEM images of TPV M0C2, TPV M5C2 and TPV M5C8, (h)The EDS map of Mg [13].

3.3. The Introduction of Plasticizer Gain

Up to now, the role of nanomaterials in the modification of SMPC has attracted more and more attention in material engineering. Nanofillers such as carbon nanotubes and graphene can significantly improve the transition temperature of composite materials [2]. However, SMPC products do not necessarily need to increase the T_g of the material under special conditions such as human contact. Nanofillers limit the movement of polymer molecular chains and increase T_g [14]. Therefore, as shown in Fig. 6, the addition of plasticizers in SMPC can reduce the overall transition temperature of the material. The Joule heating method reduces the conductivity required to reach the transition temperature, and the electro-induced shape memory effect is improved [15].

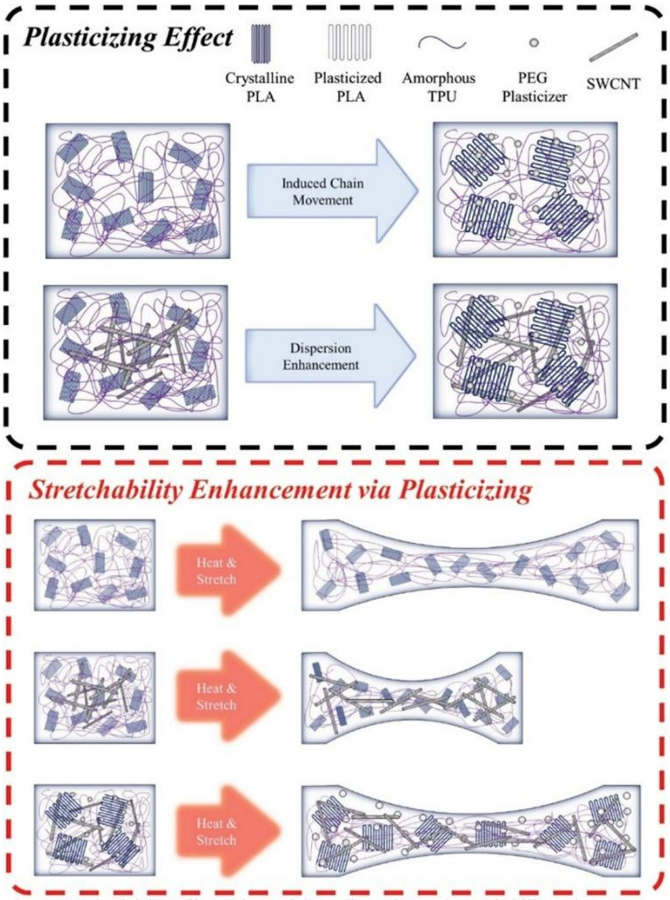

Figure.6. Effects of PEG plasticizer on PLA/TPU/CNT chain motion, nanofiller dispersion and mechanical stretching [15].

For example, Sun et al. [15] prepared a mixed electroactive SMPC that can be deformed at room temperature. For this material, thermoplastic polyurethane and polylactic acid as shape memory materials not only added the conductive material of single-walled carbon nanotubes but also mixed with polyethylene glycol as a softener. In order to make the material maintain good performance, the amount of carbon nanotubes needs to be controlled. The addition of polyethylene glycol makes the conductive material more evenly dispersed, so the conductivity of the material becomes better, and the shape is easier to fix at room temperature. Through thermogravimetric analysis and differential scanning calorimetry test, it is found that the T_g. of the mixture of polylactic acid and thermoplastic polyurethane is 57.88°C. When SMPC was incorporated into SWCNTs, T_g. increased to 58.61~ 58.65°C. It is worth mentioning that when 10wt % PEG continues to be introduced, T_g can be reduced to 40.53±42.92°C.

In summary, the addition of plasticizers is effective in reducing T_g of SMPC. Therefore, the plasticizers can promote melt processing of SMPC and expand the commercial application range of related products without worrying about the thorny problem of thermal decomposition waiting for the recovery of SMPC [2]. The addition of plasticizer is a highly gainful nano-SMPC modification method.

4. Application of Electroactive SMPC

The unique properties of electroactive SMPC make it a leading candidate for many applications. The high flexibility, remote driving ability, biocompatibility and electrical conductivity of electroactive SMPC have promoted the wide application of related products in the fields of automation equipment and robots, biomedical and advanced treatment equipment, and flexible electronic materials [2].

For example, Li et al. [16] have developed a new composite membrane, which combines polycaprolactone and polyethylene glycol, and adds an aniline trimer. It can not only resist bacteria and oxidation but also change the shape under electrical stimulation. For example, it plays a role in wound treatment, because the aniline trimer makes the material react to electricity, and then polycaprolactone and polyethylene glycol together make the material more adaptable to the biological environment and become stronger. Compared with commercial products of non-electroactive wound dressing films, these films exhibit excellent biocompatibility, ability to accelerate wound healing, electroactivity, good shape memory properties and mechanical strength, as well as suitable hydrophobicity and swelling ratio. It can be seen that electroactive SMPC is particularly suitable for biomedical and micro-technology because of its easy control, remote operation and fast response [2].

In addition, the characteristics of electroactive SMP match the development of flexible electronics. People prefer SMPC with good softness, high flexibility and high ductility. For example, Xia et al. [17] have developed an electroactive SMPC based on Eucommia Ulmo Ides Gum (EUG) as a polymer platform and CNT as a nanofiller. As a basic material, EUG innovatively shows considerable potential in enhancing the mechanical strength of the original material and the shape memory performance of the emerging electroactive field, which makes the SMPC suitable for dedicated actuators and sensors. This study shows that for every 100g of this polymer matrix containing 8g CNT in the composite material, under the condition of applying a 120V voltage, the shape recovery of SMPC can be achieved up to 93.3% in just 3 seconds. In summary, the application direction d of SMP for the preparation of flexible electronic materials such as shape memory thin film transistors and light-emitting diodes benefits from the introduction of electrical stimulation in the case of large deformation to support the photoelectric stability of related electronic products. At the same time, it also has excellent repeated bending-recovery cycle characteristics and has promising commercial application potential.

5. Conclusion

In this paper, based on the current development of shape memory polymer (SMP), the modification opportunity of SMPC to existing SMP is proposed, and the programming, recovery and material effects of shape memory behavior are discussed. Combined with typical research results, the introduction of shape memory behavior in material engineering, the preparation and modification of SMPC for the performance improvement of SMP and SMPC are analyzed. Finally, combined with the current modification of SMPC, the application advantages of SMPC in biomedicine, electronic products and other fields are briefly described.

On the whole, SMPC shows outstanding scientific research potential and commercial prospects in many aspects such as replacing SMP and promoting the development of shape memory due to a series of effective measures such as the active introduction of fillers and the development of nanocrystallization. However, people still overcome a series of problems through various ways, such as the proportion of SMPC filler, the negative change of material structure caused by programming, the influence of material properties caused by SMP turning to SMPC, and the uneven dispersion of nano-fillers in SMPC. Fortunately, as described in this paper, the current research on the performance improvement of SMPC has gradually overcome some problems, and will expand the significant advantages, rich functionalization and broad application future of high-performance SMPC through more ways. For example, the combination of 3D printing and shape memory polymers has led to the emergence of the term ' 4D printing '. The latter term indicates that a fourth dimension, namely time, is added to the 3D printing material due to the external stimulation-induced shape memory effect. The research on the performance improvement of SMPC will bring more and more development opportunities in this field.

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