Investigation of the Effect Of Morphology, Electric Field, and Cooling Regime on the Mechanical Lifetime of Isotactic Polypropylene (iPP)-based SiO_2 Nanocomposites

Hijran Ibrahimova [1*] and Rovnag Rzayev[2]

[1]Institute of Physics, Azerbaijan National Academy of Sciences (ANAS), Baku, Azerbaijan
[2]Azerbaijan State University of Economics (UNEC), Baku, Azerbaijan
`hicran90@rambler.ru`

Abstract. This study investigated the influence of incorporating nanosized silicon dioxide (SiO_2) particles at various volume fractions (1–10 vol.%) into an isotactic polypropylene (iPP) matrix on the resulting nanocomposites' morphology, mechanical properties and lifetime (τ_w). The composite samples were prepared by hot pressing, and their supramolecular structures were controlled by varying the crystallisation conditions (fast and slow cooling). Additionally, the effect of an external electric field on the material's mechanical stability and internal structure was evaluated using the electrothermopolarisability (ETP) process. Analysis based on Jurkov's theory revealed that the optimal nanofiller concentration is 5 vol.%. At this concentration, the composite's mechanical strength increased by 18%, aggregation in the structure was suppressed, and the number of active damage sites decreased. Modifying the crystallisation regime, particularly by rapid cooling, produced a more amorphous and homogeneous structure, thereby enhancing mechanical durability. Applying ETP at the optimal field intensity (10^7 V/m) maximised the lifetime by strengthening interphase interactions. Morphological and micromechanical analyses showed that combining proper SiO_2 dispersion with electrothermal treatment significantly improves the structural stability and mechanical performance of iPP-based composites. These findings suggest that such nanocomposites have potential for use in engineering applications requiring long-term performance, particularly for electrical insulation

Keywords: supramolecular structure, fast and slow cooling, spherulites, mechanical durability, activation energy

1 Introduction

In the modern era of rapid materials science development, there is an increasing need to create polymer composites with enhanced mechanical properties. Polypropylene (PP) is one of the most widely used thermoplastics and possesses several advantageous mechanical and chemical properties. This thermoplastic polymer is widely used in packaging, automotive manufacturing, electronics and construction [1, 2].

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Nevertheless, despite its many advantages, isotactic polypropylene (iPP) has limited mechanical performance, restricting its use in structural components, electronics, insulation and thermal systems. An effective approach to modifying the properties of polymer matrices is to incorporate nanofillers, which enhance interphase interactions and improve properties. The addition of nanosized silicon dioxide (SiO_2) to polypropylene significantly improves the material's mechanical properties and morphology thanks to its high dispersion, thermal stability, and interaction capability with the polymer matrix. These characteristics make it a promising component for developing next-generation nanocomposites. Silicon nanoparticles have a high specific surface area and are chemically active. This means they can substantially influence the morphology and mechanical behaviour of the polymer matrix. Previous studies on PP/SiO_2 composites have highlighted the potential and limitations of these materials. In particular, Wasel et al.'s study [3] demonstrated that incorporating SiO_2 nanoparticles into a polypropylene (PP) matrix positively affects the material's mechanical and thermal properties. This study aimed to improve not only the physical, mechanical and thermal properties, but also to enhance and effectively control the coefficient of thermal expansion (CTE). It has been reported that adding a small amount of SiO_2 filler reduces the CTE of the composite without altering the polymer structure. Analysis of another scientific study indicated that significant results were obtained regarding the effect of SiO_2 nanofillers on PP [4]. The literature reports on investigations into HHR102 polypropylene reinforced with silicon nanodispersoids of various mass fractions. Nanocomposites produced via injection moulding were tested for tensile strength, elongation, tensile modulus and impact resistance. Furthermore, the samples underwent dynamic mechanical analysis (DMA) and thermogravimetric analysis (TGA) to determine the dynamic modulus and thermal stability. Increasing the silicon content in PP nanocomposites was found to significantly improve thermal stability, with 30% and 50% lower weight losses. In the study by Liu et al. [5], basalt fibre composites modified with nano-SiO2 particles were shown to exhibit significantly improved mechanical and insulation properties. The authors showed that nano-SiO_2 enhances adhesion between the matrix and fibres, optimising electrical insulation performance and structural stability Yang Sui and colleagues [6] investigated the rheological and mechanical properties of polypropylene (PP)/silica (SiO_2) nanocomposites used in the automotive industry. They found that adding SiO_2 improves not only the mechanical strength of the PP matrix, but also the melt viscosity and rheological behaviour of the material. These results suggest that dispersing SiO_2 nanofillers in the PP matrix significantly affects both static mechanical strength and flow behaviour during processing. However, these studies have not thoroughly investigated the long-term mechanical stability of the composites or the influence of external factors such as thermal regimes and electric fields. Joraid et al. [7] studied the effect of SiO_2 nanoparticles on the melt crystallisation kinetics and mechanical properties of polypropylene. They showed that the nanofillers increased the crystallisation temperature and degree of crystallinity, thereby significantly improving tensile strength and stiffness. The study emphasised that SiO_2 particles act as nucleating agents and accelerate the matrix structuring process. Nevertheless, research into the behaviour of polypropylene (PP) and silicon dioxide (SiO_2) nanoparticle composites under the influence of electric fields is still in its early stages. Nevertheless, some significant electric-field-related properties of PP/SiO_2-based composites have been identified. Reference [8] examined the influence of nanostructured SiO_2 particles on charge trapping and electrical breakdown properties within an isotactic polypropylene (iPP) matrix under an electric field. Surface modification of the nanoparticles improved their dispersion in the PP matrix, significantly reducing the accumulation of space charges. Consequently, the electrical resistivity of the nanocomposites increased. These findings suggest that surface modification and nanoparticle content are critical parameters for optimising electric field stability. In reference [9], the impact of modifying the surface

The research methodology included morphological analysis, polarisation–depolarisation current testing and step stress accelerated ageing testing (inverse power law). The results showed that adding modified SiO_2 improved the charge trapping ability of PP. Although previous studies have demonstrated the positive effects of SiO_2 nanoparticles on the physical and functional properties of PP-based composites, the long-term mechanical stability of composites with an isotactic polypropylene (iPP) matrix, i.e. their lifetime, has not been adequately investigated. Furthermore, key factors such as the impact of crystallisation conditions (fast and slow cooling), morphological structure, activation energy and external electric fields on mechanical properties and lifetime have not been extensively addressed in the literature. In this context, the present study aims to examine the morphology and mechanical lifetime of iPP composites modified with nanoscale quartz (SiO_2) particles, as well as the influence of various physical factors such as cooling regime and electric field.

2 Experimental Section

Isotactic polypropylene (iPP) granules with a size of 5 mm (Dema Import and Export Co. Ltd., China), a density of 0.92 g/cm^3, a molecular weight of 300–700 thousand, and a specific volumetric electrical resistivity of 10^{14}–10^{15} Ω·m were used as the polymer matrix for the composites. Silica (SiO_2) nanoparticles with a particle size of 20–25 nm (Sigma-Aldrich, St. Louis, Missouri, USA), a density of 1.96 g/cm^3, and a molecular weight of 60 g/mol were used as the filler. The shapes and sizes of the nanoparticles were determined from TEM images (Fig. 1).

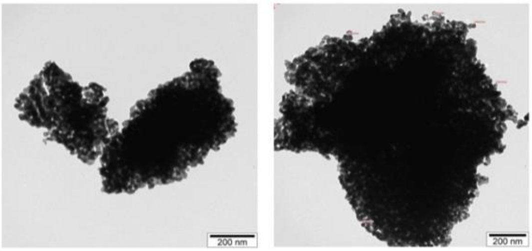

Fig. 1. TEM images of silica (SiO_2) nanoparticles

The iPP granules were dissolved in toluene at 120°C. Subsequently, the SiO_2 nanoparticles were added to the solution and stirred with a magnetic stirrer for 1 hour. The mixture was subjected to thermal treatment for 3–6 hours. To remove free solvents, the resulting solution was treated under vacuum conditions. After drying the toluene, PP/SiO_2 composites were prepared with different filler contents (1 vol%, 3 vol%, 5 vol%, 7 vol%, and 10 vol%).Disc-shaped composite samples were obtained by hot pressing at 175°C under a pressure of 15 MPa. The pressing time after reaching the target temperature was 3 minutes. Different supramolecular structures of the nanocomposite films, with thicknesses of 70–100 μm, were achieved by varying the crystallization conditions. The sample preparation process is schematically shown in Figure 2

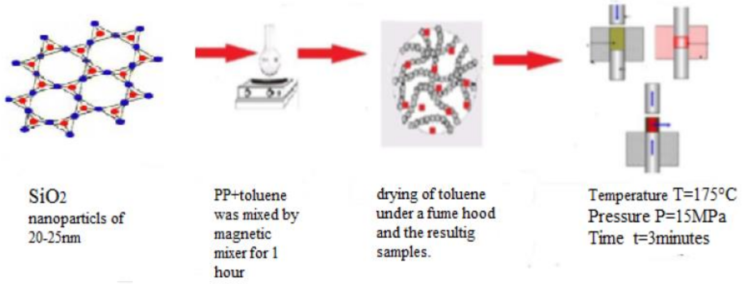

Fig. 2. Technology for the production of polypropylene doped with SiO_2 nanoparticles

For this purpose, the molten nanocomposites were cooled at different cooling rates ranging from 2 to 20 °C/min. During the rapid cooling mode, the molten nanocomposite was immersed in ice water, achieving a cooling rate of approximately 20 °C/min. In the slow cooling mode, the molten nanocomposite was cooled under pressure to room temperature at a rate of 2 °C/min.Elemental composition of the nanoparticles was determined using energy-dispersive X-ray spectroscopy (EDAX), performed in conjunction with scanning electron microscopy (SEM, JEOL JSM-7600F, JEOL Company, USA). The results of the EDAX analysis are presented in Figure 3.Mechanical durability of the polymer composites was measured using an ISO/R527 instrument (Zwick-Roell Group, Germany). Morphology of the nanoparticles was examined using a transmission electron microscope (JEM-1400, JEOL, Japan) at an accelerating voltage of 80–120 kV. Atomic force microscopy (AFM) studies were performed using an Ntegra Prima NT-MDT microscope (Russia).Electrothermal treatment was carried out as follows: the samples were exposed to various electric field intensities for 1 hour at 373 K. For polarization, the films are heated up to this temperature, and then they stay 1 hour and are cooled under the field.

3 Results and Discussion

Figure 3 shows the electron microscope image of the PP+5%SiO_2 nanocomposite and the analysis of its structure by X-ray energy dispersive spectroscopy (EDX). The obtained substance consists of spherical nanoparticles, as can be seen in the SEM image. These particles appear to be uniformly distributed throughout the sample.

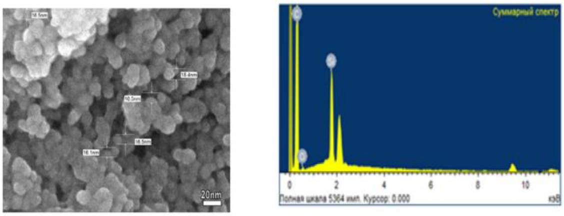

Fig. 3. Electron microscope image and structure analysis of the PP+5 % SiO_2 nanocomposite by energy-dispersive X-ray spectroscopy (EDAX).

Figure 4 shows the dependence of the mechanical lifetime (log τ_f) of PP+SiO_2 nanocomposites on mechanical stress (σ) at T = 293 K. As can be seen, increasing the nanoparticle content up to 5% improves mechanical durability. This enhancement is due to the more uniform distribution of the nanofiller and the strengthened intermolecular interactions between the nanoparticles and the polymer chains. The improvement can also be explained by changes in the size of the spherulites in the supramolecular structure, i.e. a decrease in spherulite size. The crystallites formed by the addition of the nanofiller exhibit better symmetry and consist of radially crystalline needles that form spherulites which are evenly distributed throughout the polymer matrix.

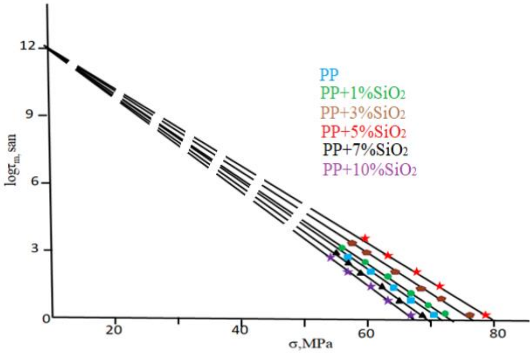

Fig. 4. Dependence of the mechanical lifetime (log τ_m) of PP+SiO_2 nanocomposites obtained under fast-cooling conditions on mechanical stress (σ) at T = 293 K.

The Jurkov theory was applied to analyse the observed dependencies. According to this concept, the mechanical lifetime can be expressed as follows:

$$\tau_m = \tau_0 \exp(\frac{U_0 \ \ -\gamma\sigma}{RT}) \tag{1}$$

The mechanical lifetime can be defined using the following formula: Here, $\tau_{\_m}$ is the time from application of the load to failure of the material. According to the literature, the value of τ_0 is constant for all materials, regardless of their chemical composition, structure, or type of bonding, and typically ranges from 10^{-12} to 10^{-14} s [10]. This value corresponds to the period of atomic vibrations around equilibrium positions in solids. U_0 is the activation energy of mechanical failure. This depends on the nature of the bonds that characterize the strength of the sample. It is also considered one of the key micro parameters that describe the failure mechanism of composites under mechanical stress.

σ is the applied stress

R is the universal gas constant.

γ is the structure-sensitive coefficient.

T is the temperature at which the analysis is performed. This expression can be rewritten by assuming a constant temperature:

$$\tau_M = Aexp(-\alpha\sigma) \tag{2}$$

Here

$$A = \tau_0 exp\frac{U_0}{RT} \text{ və } \alpha = \frac{\gamma}{RT} \tag{3}$$

Therefore, the effect of mechanical stress on lifetime can be expressed as follows:

$$lg\tau_M = lgA - \alpha\sigma \tag{4}$$

However, as the amount of SiO_2 increases (for example, by 7% or 10%), the particles tend to aggregate, disrupting the homogeneity of the composite material and consequently reducing its strength. According to Jurkov's theory, the strength of a composite material is related to micro-damage and the accumulation/release of energy within its internal structure. This theory suggests that polymer composites have a shorter lifetime as micro-damages occur. Conversely, when the composite structure is homogeneous and stable, it resists damage more effectively. Therefore, it can be said that in the PP + 5% SiO_2 composite, the number of active damage sites decreases, thereby extending the material's 'lifetime'. However, as the SiO_2 content increases (to >5%, for example), aggregations form between particles, creating localised stress concentrations. Consequently, micro-damage occurs more rapidly, a phenomenon explained by Jurkov's theory as an acceleration of micro-damage kinetics. Taking the PP + 5% SiO_2 composite as the optimal example, the activation energies for the matrix and composites with various filler fractions can be calculated using the following formula:

$$U(\sigma) = 2.3\, R\frac{\Delta(lg\tau)}{\Delta(\frac{1}{T})} \tag{5}$$

Figure 5 shows the dependence of the activation energies (ΔU) of PP and PP+SiO_2 nanocomposites on mechanical stress (σ) at temperatures of 293, 223 and 203 K. Analysis of the graph shows that the straight lines extend to approximately the same value of activation energy. The invariance of the activation energy suggests that the degradation mechanisms remain similar. This means that despite the addition of nanoparticles, which can alter the physicochemical properties of the composite, the primary degradation mechanism essentially remains the same. In other words, if the nanoparticles do not affect the chemical bonds within the polymer chains, they do not disrupt the polymer chain structure itself, but only modify the composite's other physical and chemical properties, such as its mechanical characteristics. When a mechanical load is applied, the polymer chains deform under stress. Under mechanical stress, the atoms in the polymer chains begin to shift. If the applied load exceeds the strength of the chemical bonds between atoms, these bonds can break, leading to the rupture of the polymer chain. This typically occurs in the amorphous regions of the polymer, where the chains are not well aligned and the molecules can move relative to each other.

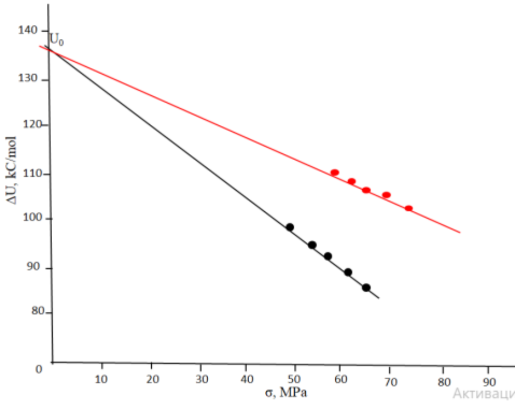

Fig.5. Dependence of the activation energy U_0_on mechanical stress (σ) at different temperatures (293 K, 223 K, 203 K) for 1-PP and 2-PP+5% SiO_2 nanocomposite

Figure 5 shows the dependence of the activation energy (U_0) on mechanical stress (σ) at different temperatures for PP and PP+5% SiO_2 nanocomposites. It can be seen that the mechanical strength of the nanocomposites increases at lower temperatures. This can be explained by the weakening of thermofluctuation processes at low temperatures [11]. Mechanical degradation occurs differently at high and low temperatures. At elevated temperatures, it is associated with structural relaxation. In low-temperature regions, the rate of structural relaxation decreases and the relaxation of the internal degrees of freedom of the molecules occurs. This can also be related to the decreased mobility of the polymer chains. In other words, the kinetic energy of the molecules decreases at lower temperatures, which reduces the movement of polymer chains relative to each other overall, thereby slowing down the development of mechanical degradation.Table 1 presents the main parameters of the mechanical properties of PP and PP+SiO_2 nanocomposites.

Table 1 Basic parameters of mechanical properties of polypropylene (PP) and nanocomposites PP+SiO_2.

Samples	**σ, MPa (τM=1 s)**	**U0, (kC/mol)**	**γ, (kC/mol)· MPa-1**	**τ0, (s)**	**τM, s (σ=65MPa)**
Pure PP	68	137	2.28	10-12	7,8
PP+1% SiO2	70	137	2.1	10-12	40
PP+3% SiO2	75	137	1.9	10-12	400
PP+5% SiO2	80 (18%)	137	1.7	10-12	1000
PP+7% SiO2	78	137	1.85	10-12	800
PP+10% SiO2	72	137	1.95	10-12	50

Table 1 shows the key mechanical properties of PP and PP+SiO_2 nanocomposites. As can be seen from the table, the parameters reach their maximum values at a SiO_2 nanoparticle content of 5%, while the U0U0U0 and τ0\tau_0τ0 values remain unchanged. The table also shows that, even under a load of 65 MPa, the 5% nanocomposite has the longest lifetime. However, the activation energy remains constant across all filler concentrations. According to Jurkov's theory, the constancy of the material's internal potential energy barrier (U_0) indicates that the energy required to initiate micro-damage does not change. A change in the structure-sensitive coefficient is observed depending on the amount of nano-filler added. This coefficient reflects the variation in stress experienced by the chemical bondsbetween macromolecules due to the presence of SiO_2 nanoparticles rather than any alteration to the bonds themselves. A small value of this coefficient combined with high mechanical strength indicates a uniform distribution of interatomic stress. In other words, as the nanoparticle content increases, the distribution of external mechanical stress among the macromolecules changes. The correlation between the structure-sensitive coefficient and the applied mechanical stress is clearly visible in the obtained graph

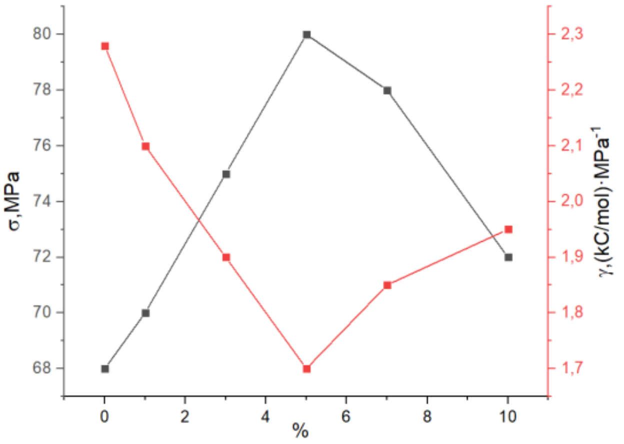

Fig. 6: Dependence of mechanical stress (σ) and structure-sensitive coefficient (γ) on nanoparticle content in PP+SiO_2 nanocomposites.

The supramolecular structure of polymer nanocomposites describes the arrangement and distribution of molecules within the polymer matrix, representing an organised system formed by the interaction between the matrix and the nanoparticles. The studies involved the use of different temperature crystallisation regimes to modify the supramolecular structures of the nanocomposites.Figure 6. Dependence of mechanical stress (σ) and structure-sensitive coefficient (γ) of PP+SiO_2 nanocomposites on nanoparticle content.

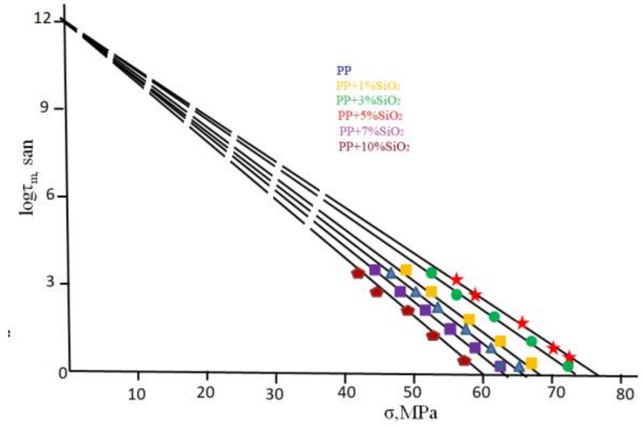

Fig. 7: Mechanical lifetime (log τ_u) versus mechanical stress (σ) for slowly cooled PP and PP+SiO_2 nanocomposites at T = 293 K.

This figure illustrates how the mechanical lifetime (log τ_f) depends on the mechanical stress (σ) for slowly cooled PP and PP+SiO_2 nanocomposites at T = 293 K. Compared to the rapidly cooled samples shown in Figure 4, it demonstrates that fast cooling positively affects the mechanical strength of the composites. During slow cooling, large spherulitic structures form within the polymer matrix. These crystalline regions promote the accumulation of internal stresses and the initiation of cracks, resulting in a decrease in the mechanical lifetime and strength of the material. In contrast, fast cooling either prevents the formation of spherulites or results in very small, weak ones. This leads to the formation of a more amorphous and homogeneous structure [12]. Consequently, the material's resistance to stress and damage increases. These characteristics contribute to higher mechanical strength and an extended lifetimeDuring production, polymers and polymer nanocomposites are exposed to various external factors, such as humidity, temperature, electric fields, radiation and electrothermal polarization (ETP) [13, 14, 15]. Prolonged exposure to these factors can significantly impact their operational properties. Figure 8 shows the relationship between the mechanical lifetime (log τ_f) and mechanical stress (σ) for the PP+5% SiO_2 composite following ETP at various field intensities. As can be seen, the mechanical lifetime reaches its maximum at an electric field intensity of 10^7 V/m; higher intensities result in a decrease. Applying ETP at an optimal field intensity of 10^7 V/m enhances molecular orientation in the polymer matrix and strengthens interfacial interactions with the nanoparticles, thereby improving the composite's mechanical strength. However, exceeding this optimal intensity can lead to microstructural damage and inhomogeneities, resulting in uneven stress distribution and a reduction in mechanical lifetime

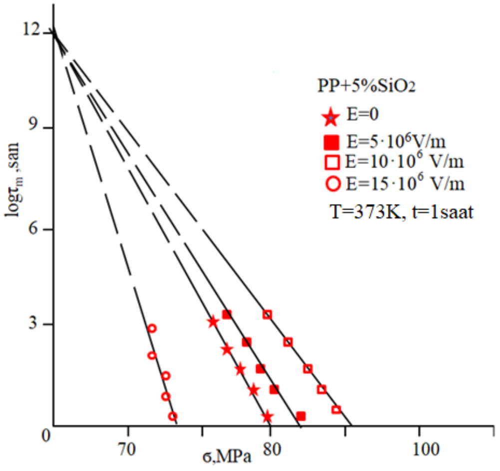

Fig. 8: Mechanical lifetime (log τ_f) dependence on mechanical stress (σ) for the PP+5% SiO_2 composite after ETP at different field intensities.

Although the ETP process does not induce dipole orientation in pure polypropylene (PP), it affects the polar regions at the interfaces in PP+silica (SiO_2) nanocomposites, leading to reorganisation at both the molecular and microstructural levels of the composite. Applying ETP at an optimal field intensity (10^7 V/m) was found to enhance mechanical strength, whereas higher field intensities reduced strength due to structural damage and crack formation. Figure 9 (a, b, c and d) shows AFM images of the PP + 5% SiO_2 composite obtained under various crystallisation conditions after 1-hour ETP exposure at $E = 10^7$ V/m and $E = 1.5 \times 10^7$ V/m. During slow cooling, larger and better-organised crystals are formed, leading to less surface chaos and greater regularity [16, 17]. AFM analyses indicate that, after one hour of ETP exposure at an electric field intensity of 10^7 V/m, the distribution of nanoparticles becomes more homogeneous, with clear alignment in surface morphology and increased orientation of macromolecules [18, 19]. This visually confirms the positive effects of the electric field and temperature on the composite structure over a short period. However, after one hour of ETP at a higher field intensity of $E = 1.5 \times 10^7$ V/m, the resulting images show structural disruption and an irregular, disordered surface

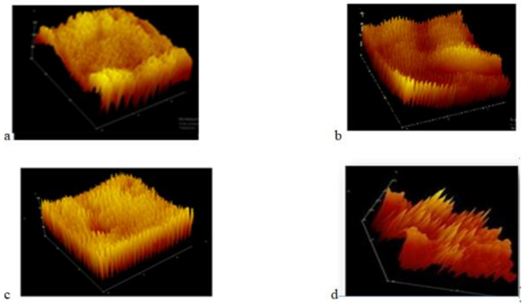

Fig. 9 (a, b, c and d) shows AFM images of the PP + 5% SiO_2 composite obtained under different crystallization conditions (a –RC and b – SC) after 1-hour electrothermopolarization (ETP) at $E = 10^7$ V/m (c) and $E = 1.5 \times 10^7$ V/m (d).

4 Conclusion

This study systematically investigated the mechanical and morphological properties of composites prepared by incorporating nanosized SiO_2 particles into an isotactic polypropylene (iPP) matrix. Significant improvements in mechanical strength and lifespan were observed when the volume fraction of nanofillers increased up to 5%. These enhancements are attributed to the uniform distribution of the nanoparticles and strengthened macromolecular interactions. The study also examined how different cooling regimes and the electrothermal polarization (ETP) process affect the structure and properties of the composite. Rapid cooling promoted the formation of an amorphous and homogeneous structure, while optimal ETP conditions facilitated a more orderly distribution of nanoparticles and molecular orientation. Together, these factors significantly enhanced the material’s mechanical durability. Analyses based on Jurkov’s theory revealed that, although the activation energy remained unchanged in the presence of nanofillers, both the structure-sensitive coefficient and the mechanical lifespan varied. This suggests that SiO_2 nanoparticles affect the microstructure and physical properties of the polymer rather than its chemical composition. The results of this study lay the groundwork for the targeted control of composite properties and open up new possibilities for improving the mechanical stability of iPP-based nanocomposites.

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