Polish Society of Composite Materials
Polskie Towarzystwo Materiałów Kompozytowych

21: 4 (2021) 141-148

COMPOSITES THEORY AND PRACTICE
ISSN: 2084-6096
ISSN (online): 2299-128X

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Received (Otrzymano) 22.09.2021

DISTRIBUTION OF MICROHARDNESS IN POLYPROPYLENE/TALC MICROCOMPOSITE

In this study the distribution of microhardness in a polypropylene microcomposite reinforced with talc microparticles was measured experimentally. The microhardness was measured at different points of the composite material to try to observe the effects of the talc particles and their proportion in the composite on the hardness of the reinforced polymer. Four proportions of talc were used: 5, 40 and 50 wt.%, in addition to virgin polypropylene, which was taken as the reference. Statistical analysis was performed on the distribution of the microhardness in the PP+talc composites to determine the average microhardness and the standard deviation. The obtained results reveal a random distribution of the microhardness of the composite, but in general the presence of talc particles increases the microhardness of the polypropylene.

Keywords: polypropylene, talc, microcomposite, micro hardness, tensile test

INTRODUCTION

Polypropylene (PP) is one of the most widely used thermoplastic polymers in the industry with a fairly good price/performance ratio. Polypropylene ranks third among polyolefin in terms of use. Polypropylene is synthesized by polymerizing high purity propylene gas from gases produced in olefin plants and oil refineries. Polypropylene is characterized by excellent electrical, insulating and moisture barrier properties [1-5]. This material can easily be reinforced; however, it has low hardness and a low operating temperature [6-9]. Researchers have introduced reinforcement to improve several of the properties of PP such as its dimensional stability, barrier and membrane separation properties, flammability resistance, strength and thermal stability. To improve the physical properties of polypropylene such as impact and heat resistance, formability, flexibility, manufacturability and resistance to cold and other atmospheric conditions, it is usually compounded and combined with different minerals such as talcum powder, calcium carbonate, or other compounds such as glass fiber and EPDM [10, 11].

Talc is a hydrated magnesium silicate (in sheets) with the formula $Mg_3Si_4O_{10}(OH)_2$. The basal surfaces of this elementary sheet do not contain hydroxyl groups or active ions, which explains the hydrophobic and inert character of talc. Talc is practically insoluble in water, weak acids and bases. It is neither flammable nor explosive. Despite its very low chemical reactivity, talc has a marked affinity for certain organic chemicals; it is indeed organophilic. Above 900°C talc gradually loses its hydroxyl groups and above 1050°C it recrystallizes into various forms of enstatite (anhydrous magnesium silicate). The melting point of talc is 1500°C. Talc is one of the most widely used reinforcements to improve the strength of PP. Talc offers many advantages to polypropylene such as enhanced stiffness and dimensional stability in automotive parts (under hoods, dashboards, inside bumpers and exterior trim) and in other industrial applications. Advanced grinding technology is required to obtain the finest talc without diminishing the reinforcing power of its lamellar structure

The PP-talc combination has been the subject of several studies [12-15]. In addition to a positive effect on the mechanical properties, talc particles also have a beneficial effect on the macromolecular orientation of PP. The PP+talc composite is chemically inert and water repellent, so its use in food or cosmetic packaging can be very advantageous. Concerning the mechanical properties, it has been shown that talc particles improve the impact resistance of PP when mixed with EPDM elastomer [6].

In this study we present experimental results concerning the tensile and microhardness tests of polypropylene specimens reinforced with different proportions of talc (5, 40 and 50 wt.%). The aim is to analyze the effects of the reinforcement on the distribution of the microhardness in the PP+talc microcomposite.

PREPARATION OF MICROCOMPOSITES

The PP used in this study is Adstif HA840R, a homopolymer that has extremely high stiffness and a high gloss. Adstif HA840R was specially developed for the production of injection molded items for which high stiffness is required. The properties of this PP include high crystallinity, high stiffness and good dimensional stability. Adstif HA840R is applied in the manufacture of food packaging containers, household items, small appliances and technical parts. No nucleating agents have been added to this polypropylene. The main physical characteristics of the PP are: 0.9 g/cm^3, melt flow rate (230°C/2.169 kg): 20 g/10 min. The Vicat softening temperature of this PP is about 158°C. A Romer injection molding machine was employed to develop the microcomposite. In order to improve the properties of the PP, talc was added with different contents ranging from 5 to 50% by weight. A fine powder was obtained from the original talc powder with a density of 2.78 g/cm^3. Sedimentation analyses revealed an average particle size of 1 µm. X-ray energy dispersive spectrometry (EDS) was used to analyze the talc particles reinforcing the PP. The chemical compounds detected were $CaCO_3$ (93%), SiO_2 (4%), MgO (2%) and Pt (1%).

EXPERIMENTAL SETUP

Tensile test

Uniaxial tensile tests were performed on a Zwick-Roell universal testing machine with a capacity of 25 kN, equipped with a test speed control system (Fig. 1). A video extensometer was used to measure the strain variation during the tensile test. All the tests were performed at the speed of 5 mm/min.

Fig. 1. Tensile testing machine

Tensile test specimens of the PP and PP+talc with different proportions of the reinforcement were made by injection to ensure good dispersion of the talc in the PP. The tensile tests were performed according to the NF EN ISO 527 standard. The dimensions of the specimens are shown in Figure 2a.

Microhardness test

Vickers hardness tests were performed with an FM-ARS9000 (Fully-Automatic Microhardness Testing System, Future-Tech Corp., Tokyo, Japan) as seen in Figure 3. The microhardness test was performed on flat specimens of dimensions 120x10x2 mm. We took measurements at five points in each specimen on a total of five specimens, which gives the number of measurements of 25 for each proportion of talc (Fig. 2b).

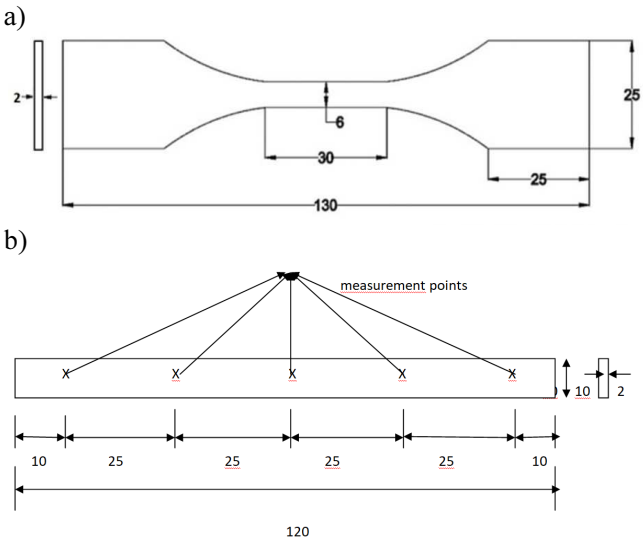

Fig. 2. Dimensions of specimens: a) tensile test specimen (size in mm) b) microhardness specimen (size in mm)

Fig. 3. Microhardness tester

The test consists in imprinting the specimen with an indenter in the shape of a pyramid with a square base under a load of 300 g for 10 s. The diagonal "d" of the imprint left on the surface of the specimen after removal of the load is measured (Fig. 4). The microhardness tester is connected to software that instantly gives

the value of the microhardness. The microhardness was measured at different points on the PP+talc specimens with different talc contents (0, 5, 40, and 50 wt.%).

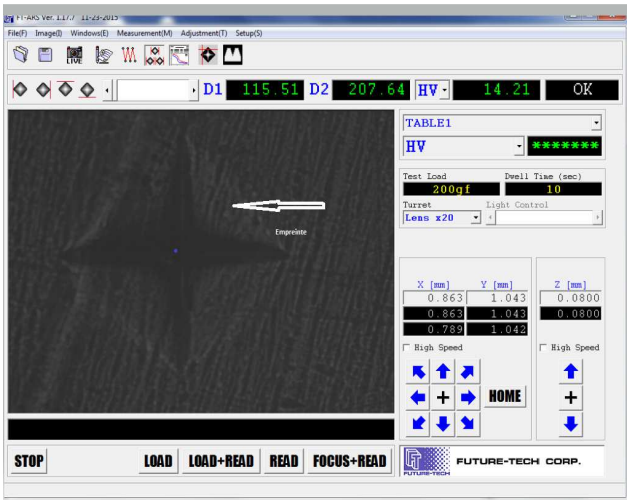

Fig. 4. Imprint left by indenter

RESULTS AND DISCUSSION

Tensile tests results

From the tensile test, we plotted the stress-strain curves of the pure PP and microcomposites (PP+talc) with different proportions of reinforcement. The mechanical properties of the composite such as the modulus of elasticity, ultimate stress and ultimate strain were deduced from the stress-strain curves. Figure 5 shows the stress-strain curves at room temperature of the PP+talc composite for the different filler weight contents: 5, 10, 40 and 50%. We also presented the curve of the pure PP as a reference. From this Figure, it can be seen that the presence of talc in the polymer has significant influences on the mechanical properties. We can confirm that all these properties are affected by the presence of the talc particles, especially the ultimate d strain. Indeed, this property is strongly reduced by the presence of the talc particles. For example, the ultimate deformation of the pure PP is about 0.227, whereas the presence of 5 wt.% talc reduces this property by 17%. With 50 wt.% talc, the ultimate strain is reduced by 57% (Fig. 5).

On the other hand, and still according to the results in Figure 5, the presence of talc reduces the ultimate stress of PP but the rate of this reduction depends on the talc content. This rate is less important than the one recorded for ultimate strain. The reduction in ultimate stress and strain by the talc is mainly due to the high stiffness of the talc particles. We can conclude that the plasticity of the polymer is significantly reduced by the talc particles. According to Figure 6, the elastic modulus of PP is increased by the talc particles, which confirms that the stiffness of PP is improved by the addition of this reinforcement. The PP rigidity increases when the talc content increases as well.

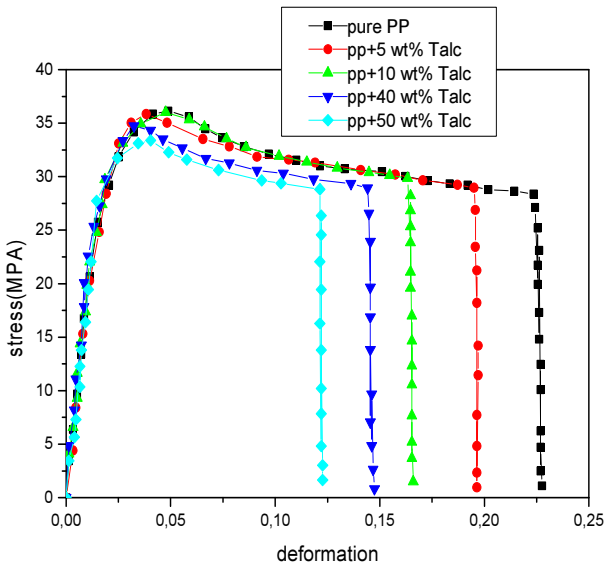

Fig. 5. Stress-strain curves of pure and filled PP

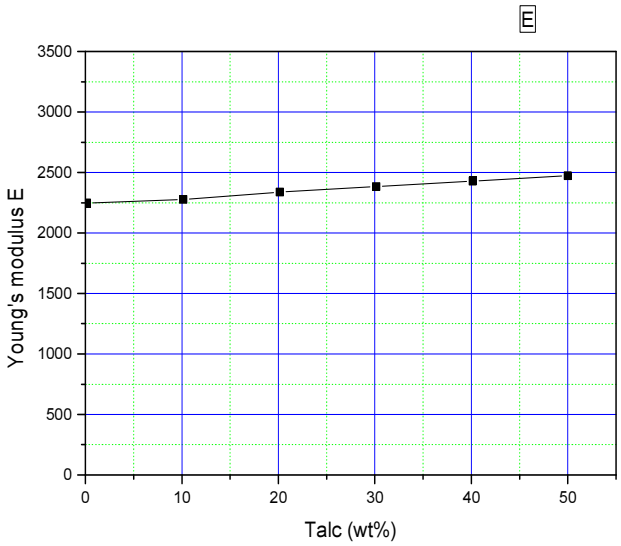

Fig. 6. Young's modulus vs talc content

Microhardness results

Pure PP

Vickers microhardness tests were performed on a flat specimen of pure PP with the dimensions 100x6 x0.5 mm. The test was conducted on five points in this specimen, and was repeated five times (5 specimens); the results are presented in Figure 7. The dispersion of the results for the two tests can be seen; this dispersion is greater for the first point in the specimen. The maximum value of the hardness is 14.1 and the minimum value is 8.94, giving an average between the extreme values of 12.52. The dispersion of the microhardness can be explained by defects present in the material after injection molding. Indeed, the differences in temperature after injection molding can give rise to microcavities that locally result in a drop in the microhardness. Local hardening can also arise due to injection molding, which gives rise to increases in the microhardness.

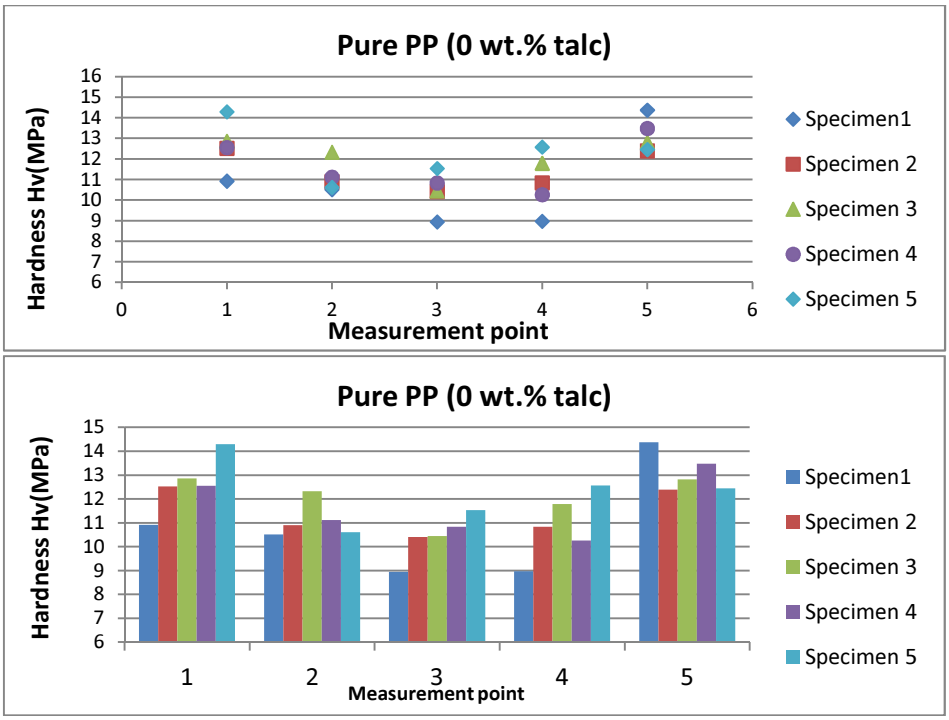

Fig. 7. Distribution of microhardness in pure PP

PP+5 wt.% talc

The same microhardness test was carried out on PP+talc specimens with 5 wt.% reinforcement. The number of tests carried out is 5, the results of which are presented in Figure 8. It can be seen from these two figures that the dispersion in the microhardness values is higher between the different measurement points compared to the pure PP.

It can be concluded that for PP+5 wt.% talc, it is very difficult to characterize the hardness of the micro-composite at the micrometric scale; it is therefore imperative to move to the nanometric scale to measure the hardness. Stochastic analysis is necessary to analyze the dispersion of the microhardness between the matrix (PP), reinforcement (talc) and the interface between the matrix and the reinforcement.

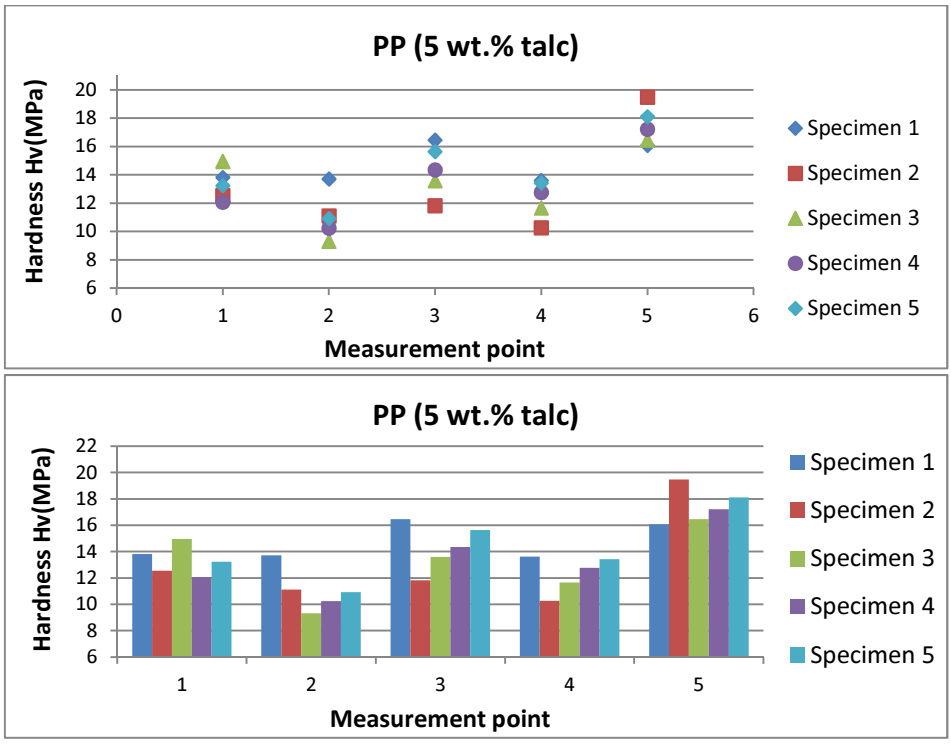

Fig. 8. Distribution of microhardness in PP+5 wt.% talc

PP+40 wt.% talc

In Figure 9 we present the distribution of microhardness in the PP+40 wt.% talc for the different samples and the different points in each sample.

It is very clear in this figure that the dispersion of microhardness between the different samples decreased compared to the cases of pure PP and PP+5 wt.% talc, but this dispersion increased between different points in the same samples. There is a large difference between the hardness of PP and talc that increases the difference between the different points in the same sample. Nevertheless, in general, we notice an increase in the microhardness of PP+40 wt.% talc compared to pure PP and PP+5 wt.% talc, confirming that talc improves the stiffness of the polymer matrix.

It can be seen that the microhardness can be measured with good approximation for this content of talc (40 wt.%), but measurement at the nanometric scale is more reliable given the size of the talc particles (3 μm).

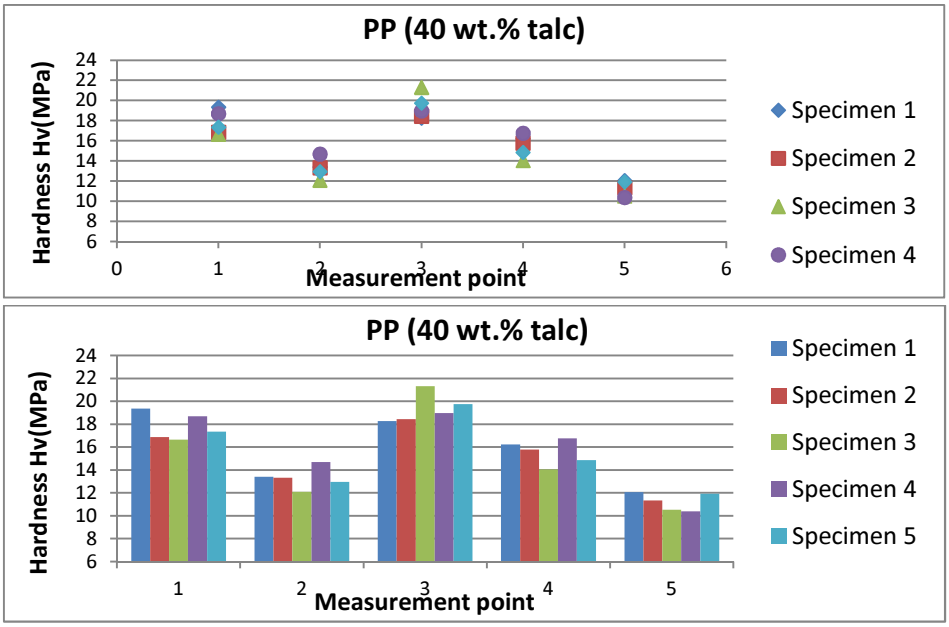

Fig. 9. Distribution of microhardness in PP+40 wt.% talc

PP+50 wt.% talc

The hardness test protocol was also carried out on five PP specimens manufactured by molding and reinforced with 50 wt.% talc (Fig. 10). We can clearly see that for this content of talc, the differences in microhardness between the specimens are not significant. This is essentially due to the relative homogeneity of the material since it contains 50 wt.% PP and 50 wt.% talc.

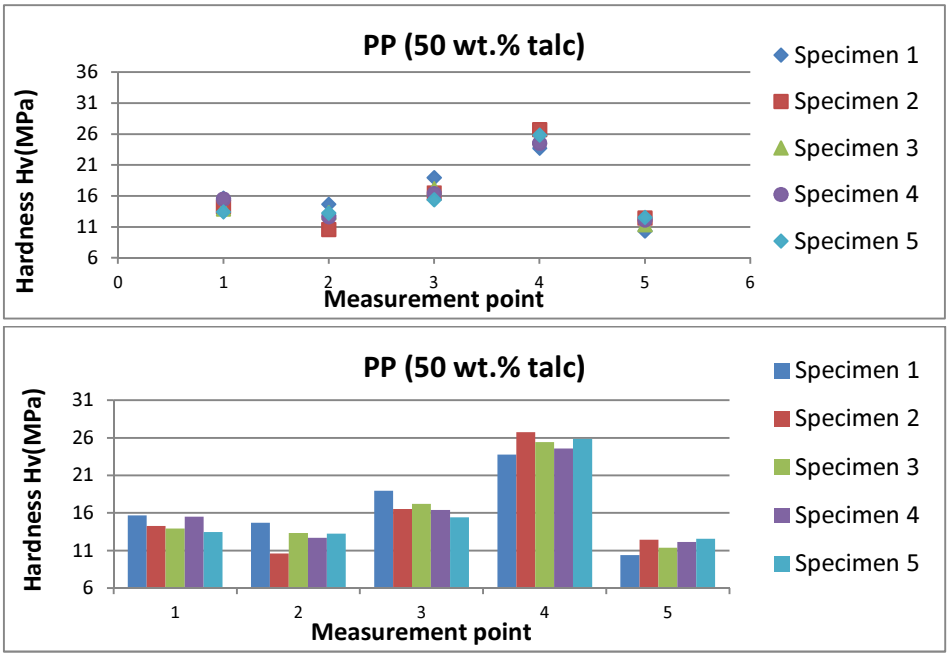

Fig. 10. Distribution of microhardness in PP+50 wt.% talc

Statistical analysis

Given the large dispersion of the microhardness values of PP with different talc contents, we considered it useful to complete the study with statistical analysis to better understand the distribution of this mechanical property in the microcomposite. In this analysis we will present the evolution of the average of the microhardness and its standard deviation for each point in the different specimens, as well as the overall average and standard deviation for the five specimens for each talc content.

Figure 11 shows the variation in the average microhardness of the different points of the five samples for pure PP (0 wt.% talc). It can be observed that the average value of the microhardness (HV) is close to 12 MPa, which is the reference value given in the literature for pure PP [12]. The exception is point 1, where the average microhardness is around 10.6 MPa.

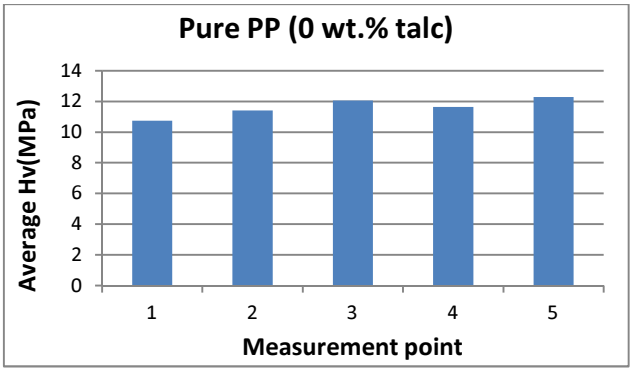

Fig. 11. Average values of microhardness at different points on pure PP specimens

This relatively low value can be attributed to the microdefects that result after injection molding. Overall, it can be stated that the average recorded microhardness is quite close to the macrohardness values given in the literature for polypropylene.

Figure 12 shows the variation in the standard deviation of the microhardness for the different microhardness measurement points on the specimens. It can be noticed that the standard deviation is relatively small in the middle of the specimens and relatively significant at the ends of the specimens. This is explained by the fact that during the injection molding of pure PP the defects accumulate at the ends of the specimens due to shrinkage of the polymer after cooling. This shrinkage creates microcavities and local hardening inside the specimen, resulting in a noticeable difference in the microhardness values. Away from the shrinkage areas (in the middle of the specimens) there are fewer defects and the standard deviation is therefore lower.

In Figure 13 we present the averages of the microhardness at the different points on the specimens composed of polypropylene reinforced with 5 wt.% talc. From this Figure, significant differences can be observed between the averages of the different points compared to the case of pure PP. Indeed, the minimum average is close to the value of the microhardness of pure PP, while the maximum average is 20% higher than the hardness of PP. This difference can be attributed to the difference between the hardness of talc and PP.

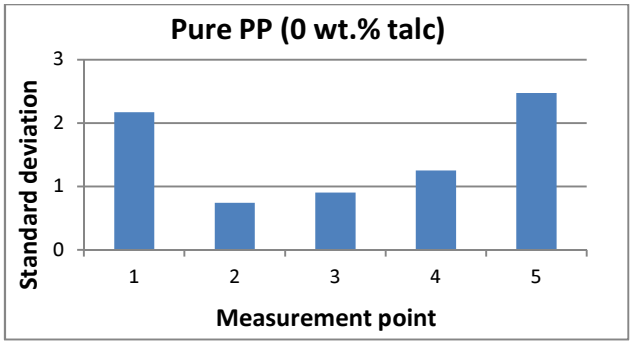

Fig. 12. Standard deviation of microhardness at different points on pure PP specimens

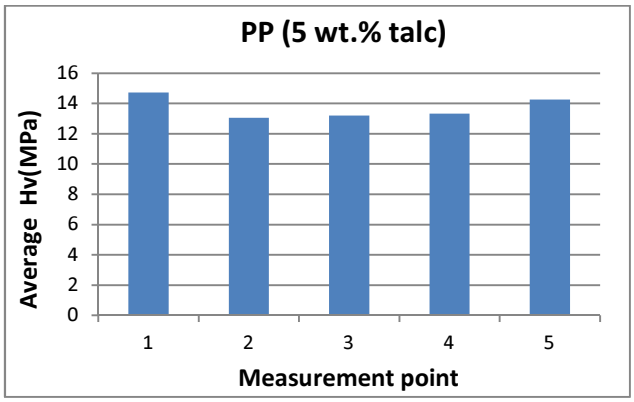

Fig. 13. Average values of microhardness at different points in PP+5 wt.% talc specimens

Figure 14 shows the evolution of the standard deviation of the microhardness at the different points of specimens composed of PP+5 wt.% talc. A clear increase in the standard deviation is noticed compared to the case of pure PP. This increase is caused by the difference between the hardness of the two components (PP and talc). Moreover, with 5 wt.% talc (a low content) the microcomposite is strongly heterogeneous, which leads to a large dispersion of the microhardness values.

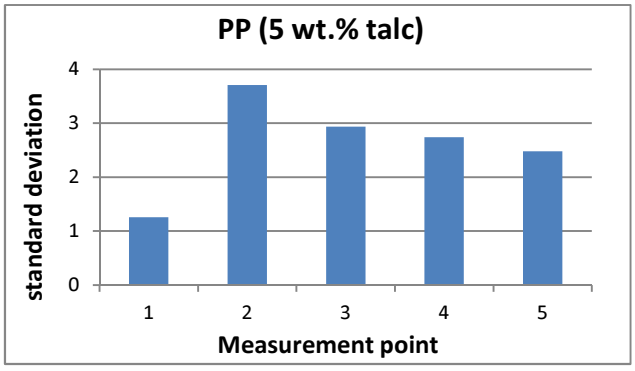

Fig. 14. Standard deviation values of microhardness at different positions on PP+5 wt.% talc specimens

Figure 15 presents the evolution of the average microhardness at the different points on the PP+50 wt.% talc specimens. It can be clearly seen that the average values of the different points significantly increased compared to those recorded for the pure PP and for the microcomposite with 5 wt.% talc. The average values for PP+50 wt.% talc are very close to the value of 16 MPa, which is the average value between the hardness of PP and that of the talc particles.

Figure 16 presents the evolution of the standard deviation of the microhardness for the different points of the PP+50 wt.% talc specimens. An increase in the standard deviation for this talc content is noticed. This increase is mainly due to the large difference between the microhardness of PP and the talc particles. On the other hand, we also note that the deviation is relatively stable with respect to the point on the specimen. This proves that with 50 wt.% talc, we have a more homogeneous microcomposite compared to the 5 wt.% talc content.

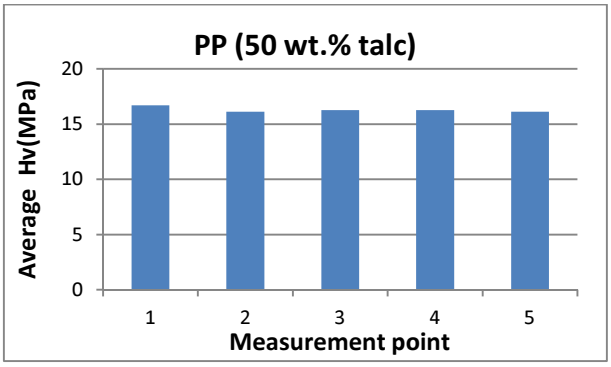

Fig. 15. Average values of microhardness at different points on PP+50 wt.% talc specimens

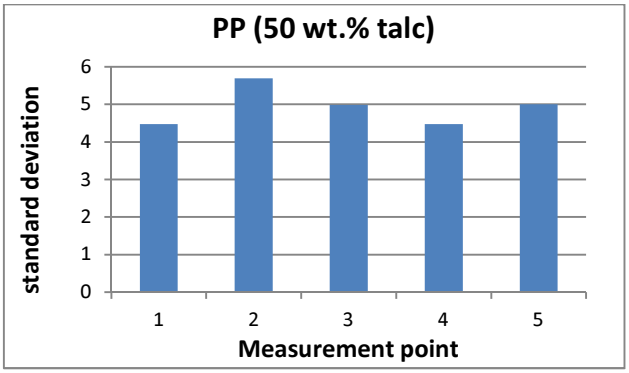

Fig. 16. Standard deviation values of microhardness at different points on PP+50 wt.% talc specimens

To complete the study, it is necessary to analyze the evolution of the average and standard deviation of the microhardness for all the specimens as a function of the talc content. In Figure 17, the variation in the global average of the microhardness as a function of the talc content is presented. An increase in this average can be observed when the talc content increases, which confirms that the addition of talc improves the stiffness of the microcomposite. Proportionality between the average of the microhardness and the proportion of talc in the microcomposite can also be noticed.

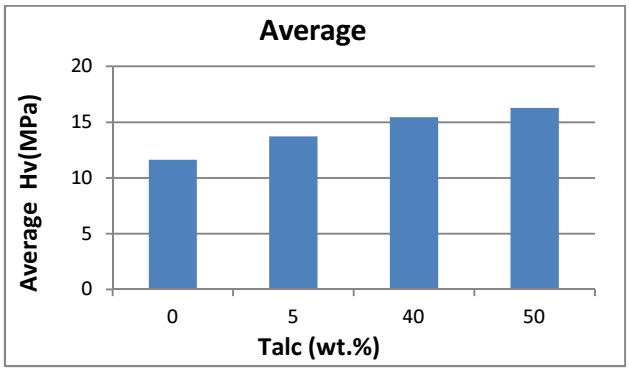

Fig. 17. Global average of microhardness vs talc content

Figure 18 shows the variation in the overall standard deviation of the microhardness as a function of the talc content. It can be seen that the standard deviation grows when the talc content also increases. We believe that the difference between the hardness of the two components of the microcomposite is the cause of the increment in the standard deviation.

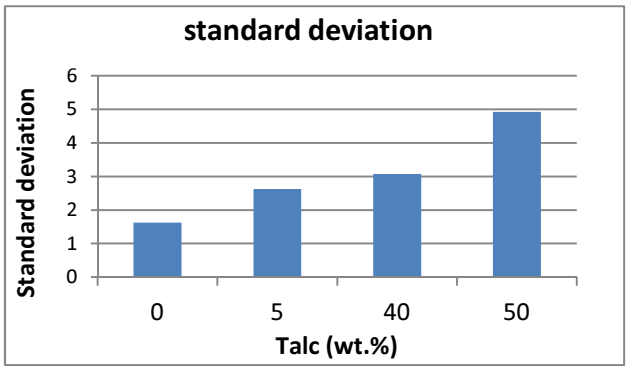

Fig. 18. Global standard deviation of microhardness vs talc content

In addition, the interfaces between the talc particles and the PP matrix increase the dispersion of the microhardness values, which necessarily leads to a rise in the standard deviation.

CONCLUSIONS

The results of this study indicate that the presence of talc particles in polypropylene improves the stiffness of the polymer while reducing its ductility. The presence of talc reduces the ultimate deformation of PP. These effects are more significant as the proportion of talc increases. It was also shown that the microhardness of PP increases when the proportion of the reinforcement also increases. However, random distribution of the microhardness in the microcomposite was found especially with an increase in the talc proportion. The presence of talc particles as well as the interfaces between the plastic and the reinforcement are the causes of the dispersions in the distribution of the microhardness.

REFERENCES

[1] Chan C.M., Wu J., Li J.X., Cheung Y.K., Polypropylene/calcium carbonate nanocomposites, Polymer 2002, 43, 2981-2992.

[2] Singer R., Ollick A.M., Elhadary M., Effect of cross-head speed and temperature on the mechanical properties of polypropylene and glass fiber reinforced polypropylene pipes, Alexandria Engineering Journal 2001, 60, 4947-4960.

[3] Carvalho G.B., Canevarolo S.V., Sousa J.A., Influence of interfacial interactions on the mechanical behavior of hybrid composites of polypropylene/short glass fibers/hollow glass beads, Polym. Test. 2020, 85, 106418.

[4] Nikmatin S., Syafiuddin A., Hong Kueh A.B., Maddu A., Physical, thermal, and mechanical properties of polypropylene composites filled with rattan nanoparticles, J. Appl. Res. Technol. 2017, 15, 386-395.

[5] Lin J.-H., Huang C.-L., Liu C.-F., Chen C.-K., Lin Z.-I., Lou C.-W., Polypropylene/short glass fibers composites: effects of coupling agents on mechanical properties, Thermal Behaviors Morphology Materials 2015, 8, 8279-8291.

[6] Maddah H.A., Polypropylene as a promising plastic: a review, Am. J. Polym. Sci. 2016, 6, 1-11.

[7] Chen J., Yan Y., Sun T., Qi Y., Li X., Probing the roles of polymeric separators in lithium-ion battery capacity fade at elevated temperatures, J. Electrochem. Soc. 2014, 161(9), 1241-1246.

[8] Jeencham R., Suppakarn N., Jarukumjorn K., Effect of flame retardants on flame retardant, mechanical, and thermal properties of sisal fiber/polypropylene composites, Compos. B Eng. 2014, 56, 249-253.

[9] Yu L., Ma Y., Loading rate and temperature dependence of flexural behavior in injection-molded glass fiber reinforced polypropylene composites, Compos. B Eng. 2019, 161, 285-299.

[10] Shubhra Q.T., Alam A., Quaiyyum M., Mechanical properties of polypropylene composites: a review, J. Thermoplast. Compos. Mater. 2013, 26, 362-391.

[11] Eiras D., Pessan L.A., Influence of calcium carbonate nanoparticles on the crystallization of polypropylene, Mater. Res. 2009, 12, 523-527.

[12] Hadal R.S., Dasari A., Rohrmann J., Misra R.D.K., Effect of wollastonite and talc on micromechanisms of tensile deformation in polypropylene composites, Materials Science and Engineering A 2004, 372, 296.

[13] Md. Gafur A., Nasrin R., Md. Mina F., Md. Bhuiyan A.H., Tamba Y., Asano T., Structures and properties of the compression-molded istactic-polypropylene/talc composites: Effect of cooling and rolling, Polymer Degradation and Stability 2010, 95, 1818-1825.

[14] Bouakkaz O., Albedah A., Bachir Bouiadjra B., Sohail MA Khan, Benyahia F., Elmeguenni M., Effect of temperature on the mechanical properties of polypropylene-talc composites, Journal of Thermoplastic Composite Materials 2018, 31(7), 896-912.

[15] Premalal H.G.B., İsmail H., Azahari B., Comparison of the mechanical properties of rice husk powder filled polypropylene composites with talc filled polypropylene composites, Polymer Testing 2002, 21, 833-839.