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Computer aided image analysis of nanocomposites microstructures

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ABSTRACT

Purpose: Scanning electron microscopy and light microscopy with polarization has been used for observation cross sectioned samples of nanocomposites with developed layered region, visible in the polarized light; Purpose of this work was to focus on investigation of nanocomposites, with polyolefin matrix and nanometric 2:1 silicate reinforcement, as future engineering materials,

Design/methodology/approach: Process of injection molding has been used to manufacture nanocomposites; conventional mold equipped with special hydraulic pressure system and connected to external computer for controlling flow movements inside mold cavity

Findings: Layered region, with medium thickness of layer equal to 200 microns, obtained due to the melt manipulation of polymer, improved fracture toughness of investigated nanocomposites; nanoparticles located in the matrix make llalom-like crack propagation extending fracture due to bypassing parallely oriented nanoclay tactoids

Research limitations/implications: Specimen shape used in the experiment is rectangular and obtained data of investigated specimens approve very good reinforcement along specimen, application of mre complicated shapes may perturb uniform distribution of nanoparticles in the matric and affect mechanical properties

Practical implications: Application developed injection moulding technique and nanomaterials gives possibility to obtain layered region with raised toughness and simultaneously obtain cheaply nanocomposites, thanks to economical technology of manufacturing and commercially available polyolefins at low price

Originality/value: Accordingly to increment of plastic market and its upward tendency, wide range of products and applications (and still increasing) in different sectors including medical, household appliances, furniture, automotive, aerospace, electronics and buildings

Keywords: Nanocomposites; Engineering materials; Polymer processing; Injection moulding

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METHODOLOGY OF RESEARCH, ANALYSIS AND MODELLING

1. Introduction

In the last decade engineers, designers and technologists try to combine many various engineering materials to keep up with the needs of economic, service and industrial sector, including energetic, metallurgical, electromechanical and chemical branches. This creates opportunities for inventors, engineers, designers and researchers to develop existing or create new materials with various structures through design of new alloys, composites and nanocomposites, using advanced manufacturing techniques, computer simulations, neural networks, artificial intelligence tools and computer aided image analysis [1-8]. One of the most significant factors influencing the attractiveness of material is specific strength (strength to its density) and smaller weight is constantly desirable. Comparison of the relative strength of selected engineering materials shows that polymer composites and nanocomposites are widely used and their demands grows (Fig. 1).

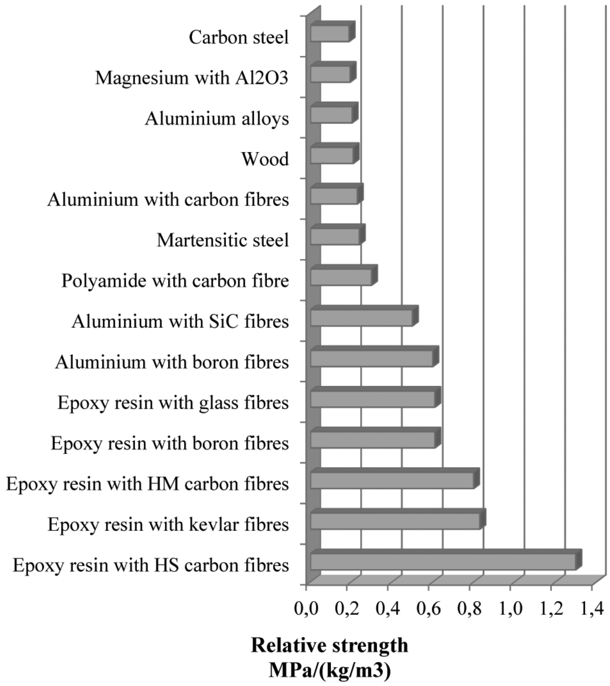

Fig. 1. Comparison of relative strength of selected engineering materials, including composites [19,20]

Additionally also polymeric materials find growing demand worldwide and prediction up to 2025 year shows increment of consumption (Fig. 2).

This confirms, that plastic market evolution will keep upward trend until 2025, reaching demand level equal to 325 million tons, which in comparison to 2013 indicates 38% increment. Accordingly to this tendency, demand for polymer composites and nanocomposites analogically will rise. Focusing on the polymer sector, can be assumed, that together with increasing consumption of dairy products, the packaging sector is mainly comprehensive. However other sectors are also involved in the use of polymers including medical, household appliances, furniture, automotive, electronics and buildings (Fig. 3) [9 -16].

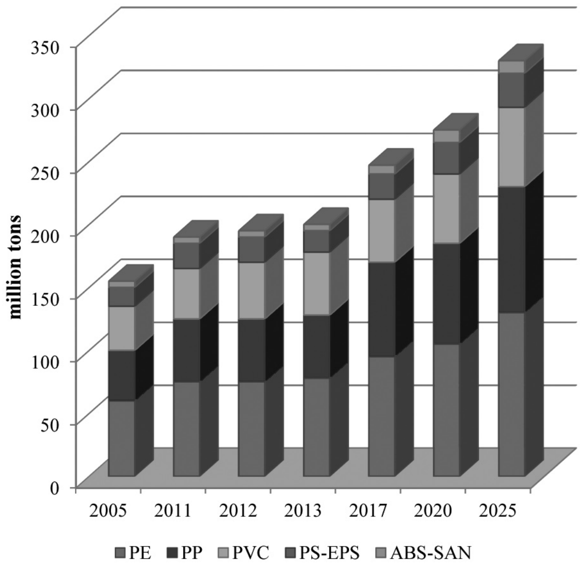

Fig. 2. World demand distribution of polymers in the years 2005-2025 [19,20]

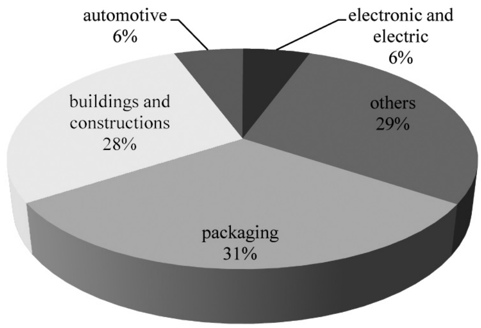

Fig. 3. Plastics application in Europe [19,20]

Within this application nanocomposites are newest materials with vision of future nanotech market, thanks to such materials like carbon nanowires, nanotubes, fullerens and quantum dots, which can provide numerous of publications, patents, company activities and scientific conferences related with nanomaterials, nano-composites, devices and nanobio [17-23].

Production of nanocomposites uses also one the most economical technique of polymers' processing - injection moulding. This process brings components which are used, among other things, in medical, aerospace, automotive and shipbuilding sectors. Injection moulding, beneficial from economical point of view, is then the process that gives possibility to produce cheap and durable materials. Manufacturers, that use injection molding techniques, are the part of designers, that direct the global development and trends in the sector of processing polymeric materials [24-30].

2. Experimental procedure

In the research has been analyzed samples of polypropylene (PP) and montmorillonite (MMT). Lis of abbreviations used in the article can be checked in the Table 1.

Table 1.
List of abbreviations used in article

	Abbreviation	Full name
Material	PP	Polypropylene (matrix)
	MMT	Montmorillonite (reinforcement)
Processing	IM	Injection Molding
	AIM	Advanced Injection Molding
	MT	Melt temperature
	IN	Interval time
	IT	Interval number

Polypropylene with melting point of 200ºC and density of 0,9g/cm^3, used in the experiment as matrix materials, has been chosen among commercially available materials with low price and easy for processing on injection machine.

Montmorillonite with melting temperature >390ºC and its density of 1,8g/cm^3, in the platelet shape in proportion 2:1, that is divided into octahedral sheet surrounded by two tetrahedral sheets, containing atoms of silica, aluminium and magnesium. Formula of the MMT is as follows: $M_x(Al_{4-x}Mg_x)Si_8O_{20}(OH)_4AlO_6$ (Fig. 4).

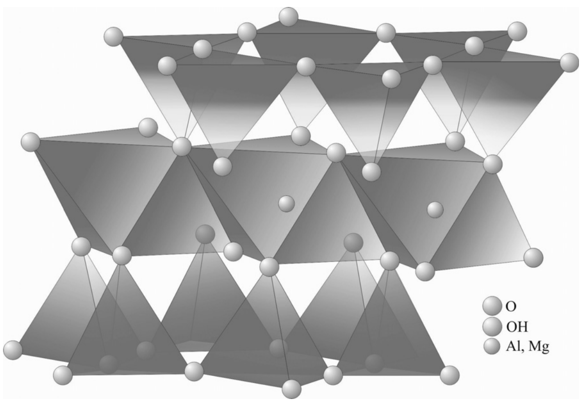

Fig. 4. Schematic illustration of atoms arrangements in a typical MMT layer

PP was mixed with MMT nanoparticles in the barrel at constant rotor speed 60 rotations per minute in the room temperature and then located in the feeder of moulding machine. Mould capacity was designed as rectangular shape, so the specimens were obtained as bars ready for fracture test (Fig. 5).

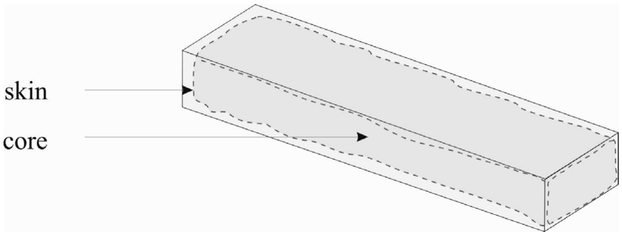

Fig. 5. Schematic illustration of specimen with visible outer (skin) and inner (core) part.

Combination between three changeable processing parameters, that is - MT, IN and IT gives totally 10 combinations - eight sets for AIM and two sets for IM. For each parameter was chosen two levels - lower and higher (Table 2).

Table 2.
Lower and higher values of parameters

Parameter	MT	IN	IT
Low value	240°C	3	1
High value	280°C	12	3

Typically for IM during injection there is creation of the skin layer, where particles are under highest shear stress comparing to the core region. (Fig. 6). This phenomenon consists on extension of particles located in outer region, closer to skin, due to strong shearing and faster cooling of material by D. Rosato and L.A. Utracki et al.

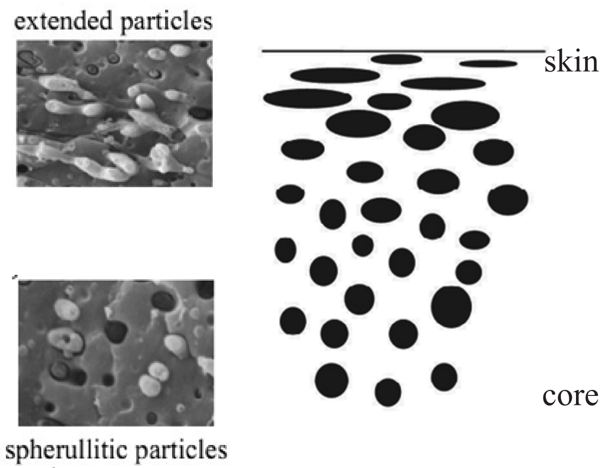

Fig. 6. Shape of particles due to different shearing stress induced during injection molding

Similar effect was achieved and multiplied in AIM process which advantage consists on creation multilayer region, due to repeating sequences of reciprocating movements inside mold until melted polymer is solidified. Confirmation of MMT particles inside polypropylene matrix was confirmed by energy dispersive spectrometry on SEM.

3. Discussion of experimental results

Study of fracture surfaces on scanning electron microscopy exposed plastic character during flexural test for specimens of pure PP performed by IM (Fig. 7) and AIM (Fig. 8).

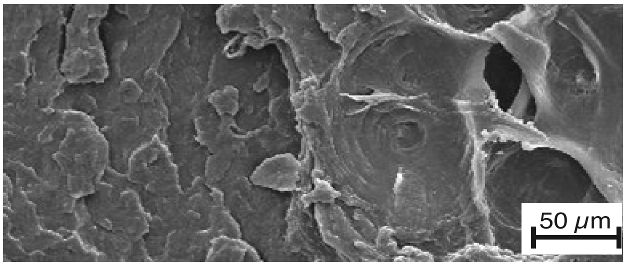

Fig. 7. Cross section of PP specimen; IM process

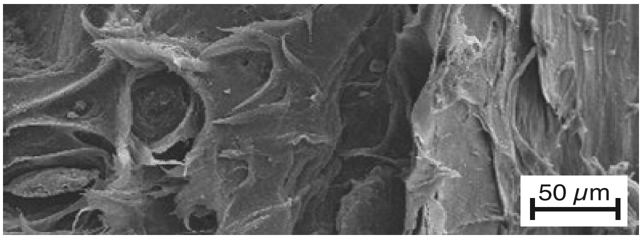

Fig. 8. Cross section of PP specimen; AIM process

Addition of 1wt% of nanoparticles didn't change the character of fracture presenting still plastic deformation. Filling of 10wt% of nanofiller showed ductile character of deformation and simultaneously weaken the mechanical properties (Figs. 9, 10).

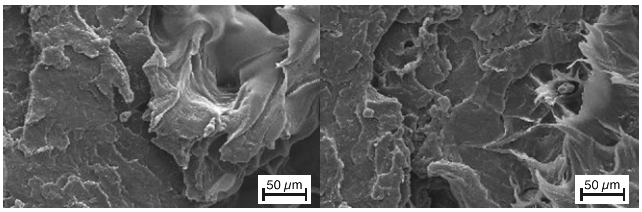

Fig. 9. Cross section of PP specimen with addition of 1wt% of MMT; IM process *(left image)*, AIM process *(right image)*.

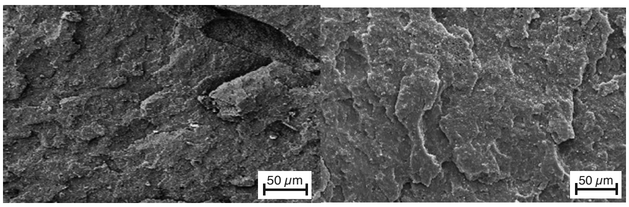

Fig. 9. Cross section of PP specimen with addition of 10wt% of MMT; IM process *(left image)*, AIM process *(right image)*.

SEM images of the fracture surfaces of the specimens could bring better look on the differences between the core, shear zone and outer skin energy absorption, by evaluation of the level of the roughness or smoothness of the surface or by calculating stress whitening area, which appears after fracture of polymeric materials. Nanoparticles influenced morphology and affect on stiffness and toughness of obtained specimens, and these properties increase together with increment of nanofiller content.

Cross section of PP specimens, performed by IM, both the low (240°C) and high (280°C) temperature, is characterized by large, spherullitic core and thin skin (approximately 300 microns). AIM process enlarge skin and layered region up to 11 layers with average thickness of 200 microns.

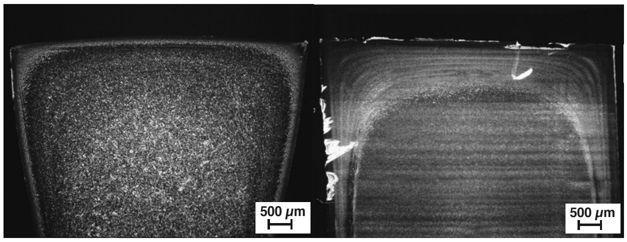

Fig. 10. Cross section of PP specimen with addition; IM process *(left image)*, AIM process *(right image)*.

Image analysis helped to calculate thickness of layers by extracting regions and layer due to use of different technique like edge detection and balancing of colour (Fig. 11).

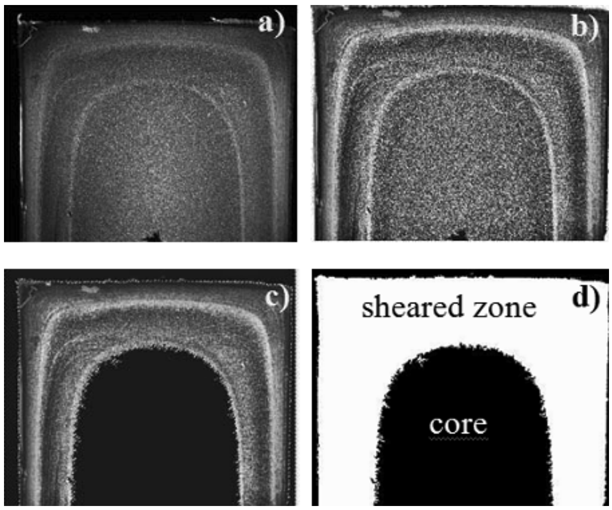

Fig. 11. Order of image analysis of cross sections of investigated specimens; a) direct image from PLM, b) contrast-brightness improvement, c) edge detection, d) mask overlapping

Basing on the extracted layers was possible to use image analysis software for calculation thickness of layers (Figs. 12, 13).

Optimization of processing, including MT, IN, IT, determining the properties, is a key issue. The graphs below show the contribution of variable parameters on flexural strength of investigated materials. Flexural strength is mainly controlled by stroke number for PP and PP with 1 and 10% of MMT (Figs. 14, 15, 16).

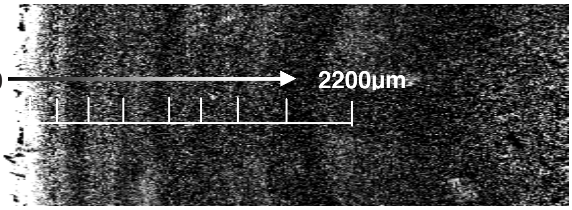

Fig. 12. Calculation of separate layers.

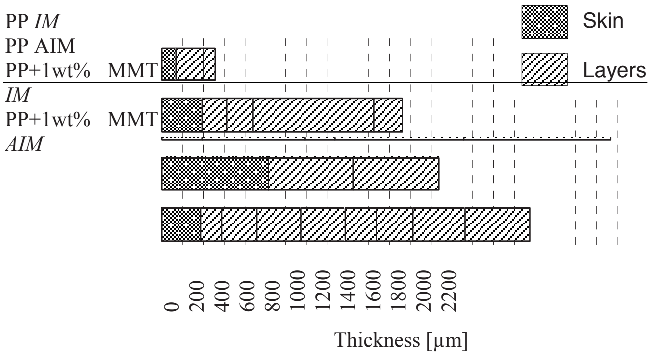

Fig. 13. Thickness of layers for PP and PP+MMT specimens obtained by IM and AIM.

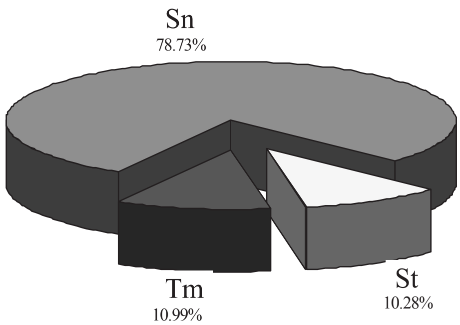

Fig. 14. Contribution of N-CIM processing parameters on flexural strength of PP; Tm - melt temperature, St - stroke time, Sn - stroke number

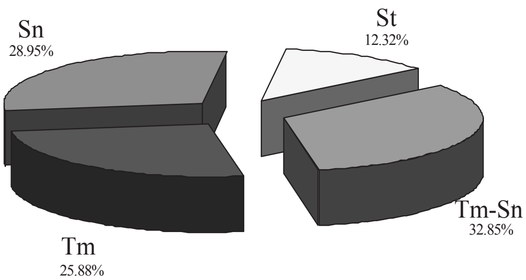

Fig. 15. Contribution of N-CIM processing parameters on flexural strength of PP/MMT 1%; Tm - melt temperature, St - stroke time, Sn - stroke number

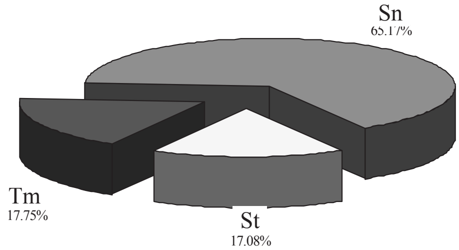

Fig. 16. Contribution of N-CIM processing parameters on flexural strength of PP/MMT 10%; Tm - melt temperature, St - stroke time, Sn - stroke number

4. Conclusions

The hydraulic pistons movement inside mold in AIM influenced on multilayer structure, showing for PP specimens, the developed region nearly 4 times more developed than in IM. Addition of nanoparticles to PP did not change extremely the dimensions of this zone, but increased the number of layers. Nanoparticles induce forming a multilayer structure, so the energy absorbed by specimen of PP with MMT was higher than in the pure PP. Addition of nanoplatelet particles is detrimental for the fracture energy of the mouldings. The AIM process created multilayer region. Specimens performed by IM contain big core, occupying 90% of specimen, which is almost double comparing to structure of specimens performed by AIM, where core occupies 52%. For the rest of specimen consist skin (IM) and shear region/skin structure (AIM). This indicates, that AIM process creates structure with developed shear zone four times bigger than skin, created in conventional injection moulding (48% to 10% ratio).

Flexural strength of PP with 1wt% of MMT 1% is controlled simultaneously by interaction between MT and IT (32.8%) and by IT (28.9%) and MT (25.8%).

MMT agglomerates can be easily splitten, due to high shear rates in the flow orientation induced by reciprocated movements. The AIM process can be proficiently used to obtain high performance nanocomposites. Creation of the layers by this process is the significant step in advancement of polymer composites and nanocomposites, simultaneously giving ability of morphology control and project materials with expected properties.

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