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Effect of Different Nanoparticles and Friction Stir Process Parameters on Surface Hardness and Morphology of Acrylonitrile Butadiene Styrene

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ABSTRACT

In the present study, the effect of material and process parameters on the morphological and hardness properties of friction stir process (FSP) acrylonitrile butadiene styrene (ABS) were investigated. For this purpose, firstly ABS polymeric sheets were injection molded. Then a slot with predetermined depth and width was created on sheets using a horizontal milling machine. Nano type (nanoclay, nano Fe_2O_3, and multi-walled carbon nanotube), rotational speed and transverse speed of FSP tool was selected as input R of the experiment in three levels. Design of experiments was carried out according to Taguchi L_9 orthogonal array. Then aforementioned three types of nano particles were added to the slots and the specimens were friction stir processed in different conditions using a simple cylindrical tool on a vertical milling machine. In the next step, the hardness tests were conducted on the FSP sections of the samples. It was found that addition of nano particles causes an increase in the surface hardness of polymeric samples. Also, it was observed that the samples with multi-walled carbon nanotubes as a reinforcement had the highest value of hardness. Scanning electron microscopy (SEM) tests were carried out on the FSP sections of specimens. Obtained SEM images indicated that processing conditions have a significant effect on the nano particles dispersion in the polymeric matrix.

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1. INTRODUCTION

Application of nanocomposites is growing fast due to their special properties. Nanocomposites are composites that at least one of their component's dimension is in the nanometer range (between 1 to 100 nm) [1-3]. In recent years, nanocomposites have shown significant improvements due to several modifications in material structures at the nanometer scale and enhancements in properties of composites [4-6].

Polymeric nanocomposites have attracted too much attention on account of their unique mechanical, physical and chemical properties. Generally, polymer nanocomposites have high strength, low weight, high thermal stability, and high chemical resistance.

Many different processes are used in order to produce nanocomposites. In most of the processes, the mixing of the base polymer and added nanoparticles is carried out by melting the polymer. However, some new methods have been proposed recently in order to make a layer of nanocomposite on polymer sheets without making the polymer melt.

Friction stir processing (FSP) has been an engrossing filed in the recent years. It is based on the principles of friction stir welding (FSW) and is used to create composite or nanocomposite layers on the polymer or metallic sheets. Mishra et al. [7] investigated the friction stir processing of SiC particles on an Aluminium sheet. The results indicated that using 27 vol% of SiC increased the hardness of sheet from 85 to 173 HV. Similar research was conducted by Morisada et al. [8] on the FSP of multi-wall carbon nanotube on an magnesium (AZ31). The microhardness was increased from 41 to 78 HV. In another research, Kumar et al. [9] investigated the FSP of SiCN particles into pure copper, increasing of hardness up to 5 fold indicated that friction stir processing is an effective way to increase

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the hardness of metals. Some more recent researches are conducted on the FSP of polymers. Baramouz et al. [10] used a new method based on FSP to produce polymer nanocomposite containing nanoclay in order to enhance distribution of nanoparticles and mechanical properties. They found out that the hardness of nanocomposite samples was increased by increasing the rotational and transverse speeds. Also, the results illustrated that nanoparticles dispersed in some regions were completely exfoliated and in some other regions were intercalated. Zinati et al. [11] studied the FSP of nanocomposite polyamide-6/multi walled carbon nanotubes (MWCNTs). They used X-ray diffraction (XRD) and scanning electron microscopy (SEM) tests to investigate the properties of nanocomposites. The results illuminated that MWCNT were distributed uniformly into the polymeric matrix. This uniform distribution enhanced the reinforcement capability of CNTs. Nakhaei et al. [12] studied tensile properties of ethylene-propylene diene monomer (EPDM)/nanoclay using FSP. They investigated the influence of rotational and transverse speeds and shoulder temperature on the tensile properties of nanocomposite samples. The results proved that by increasing rotational speed and shoulder temperature, tensile strength increases from 15.8 to 18.2 MPa while the elongation at break decreases from 46 to 22%.

According to the literature review, there was no comprehensive published research on the effect of adding different nanoparticles on the FSP sections of ABS polymer. ABS is used in the manufacturing of car interior parts and furniture. Thereby improving the surface hardness of this polymer seems to be necessary. In this research, the effect of adding three types of nanoparticles including nanoclay, nano Fe_2O_3 and MWCNT and processing conditions of FSP including rotational and transverse speeds on the hardness and morphological properties of ABS was investigated using Taguchi approach.

2. DESIGN OF EXPERIMENTS

Taguchi method is used in this research in order to investigate the effect of input parameters on the results. Taguchi approach is a combination of statistical and mathematical techniques used in experimental studies. This approach is capable of determining the optimum conditions with minimum number of experiments.

The nanoparticle type was selected as the first input parameter of this study at three levels (clay, Fe_2O_3, and MWCNT). Similarly, each of the rotational and transverse speeds of FSP tool was considered as process parameters in three levels. The input parameters and their levels are given in Table 1. According to the parameters and their levels, Taguchi L_9 orthogonal array was employed for the design of experiments using Minitab software. The list of experiments is presented in Table 2.

Taguchi method introduces a loss function that is presented as a signal to noise ratio (S/N). Depending on the problem, one of the signal to noise analysis of Equations (1) to (3) is applied.

$$S/N=10\log[\frac{\overline{Y}^2}{S^2}] \quad \text{(nominal is best)} \quad (1)$$

$$S/N=-10\log[\frac{1}{n}\sum_{i=1}^{n}\frac{1}{{y_i}^2}] \quad \text{(larger is better)} \quad (2)$$

$$S/N=-10\log[\frac{1}{n}\sum_{i=1}^{n}{y_i}^2] \quad \text{(smaller better)} \quad (3)$$

where, y is response variable, s is standard deviation and n is the number of experiments' repetitions. For each parameter, regardless of what type the problem is, the optimal level is the level in which S/N ratio has the highest value. Since in this case, higher hardness values of specimens are desirable; therefore, the “larger is better” state was used to calculate the S/N ratio [13].

3. EXPERIMENTAL PROCEDURE

3. 1. Materials and Equipment

ABS with melt flow index (MFI) of 1.7g/ 10 min (200°C, 5kg), Tabriz Petrochemical Company, was used as the base material. Montmorillonite nanoclay with trade name of Cloisite 30B, alpha- ferrous nano-oxide (α-nano Fe_2O_3) with purity of 98% and particle size of 20-40 nm and MWCNT with purity of 90%, inner diameter of 5-10 nm, external diameter of 10-30 nm and length of 10-30 µm (US reasearch nanomaterial Inc.) were used as reinforcements.

TABLE 1. Input parameters and their levels

Levels Parameters	1	2	3
Nanoparticle type	Clay	Fe_2O_3	MWCNT
Rotational speed (rpm)	500	800	1250
Transverse speed (mm/min)	25	40	63

TABLE 2. L_9 experiments of Taguchi approach

Sample	Nanoparticle type	Rotational speed (rpm)	Transverse speed (mm/min)
1	Clay	500	25
2	Clay	800	40
3	Clay	1250	63
4	Fe_2O_3	500	40
5	Fe_2O_3	800	63
6	Fe_2O_3	1250	25
7	MWCNT	500	63
8	MWCNT	800	25
9	MWCNT	1250	40

An NBM HXF-128 injection molding machine is used for injection molding of specimens. Horizontal and vertical milling machines were utilized in order to create grooves and perform FSP on polymeric sheets, respectively. Shore-D hardness test machine was employed to measure the hardness of produced FSP specimens and also the morphological properties of the produced FSP samples were investigated using a Hitachi S-4160 field emission scanning electron microscope (SEM).

3. 2. Preparation of Specimens After the design of experiments, in order to manufacture polymer sheets made of ABS, granules were dried using the dryer unit of injection molding machine at 80 °C for 24 hours. According to the processing conditions of Table 3, polymeric sheets were produced using injection molding machine in a mold with a dimension of 175×80 mm and thickness of 3.7 mm.

Then two grooves were created with a width of 1.3 mm and depth of 1.5 mm in the longitudinal direction and on one of the sides of the polymeric sheets using a horizontal milling machine. Afterwards, three types of nanoparticles were added manually into the grooves. A simple cylindrical pin was selected as the tool and the friction stir processing was carried out using a vertical milling machine and polymeric nanocomposite sheets were produced according to experiments listed in Table 2. The pin used for FSP polymeric sheets is illustrated in Figure 1. Also, Figure 2 demonstrates three different samples produced using different nanoparticles.

Finally, hardness and SEM tests of FSP polymeric specimens were conducted whose results are as follows.

TABLE 3. The injection molding processing parameters

Parameter	Value
Injection temperature (°C)	240
Injection pressure (MPa)	115
Holding pressure (MPa)	80
Holding pressure time (s)	2
Mold temperature (°C)	40

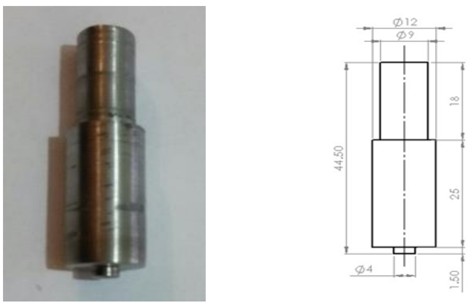


Figure 1. Pin used for FSP in this study

4. RESULT AND DISCUSSION

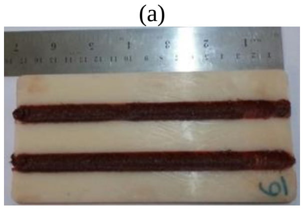
4. 1. Shore-D Hardness Test Hardness test was conducted on the produced FSP polymeric samples and in order to increase the accuracy of hardness test, specimens were tested at least on five points and the average value was reported as the final hardness result of FSP polymeric samples. Results of the hardness test are given in Table 4.

The main effects of considered parameters on Shore-D hardness are obtained using Minitab software and are presented in Figure 3.

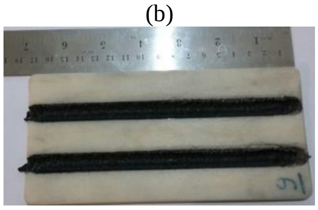
According to Figure 3, the hardness of FSP polymeric samples increases by changing the types of nanoparticles from nanoclay to nano Fe_2O_3 and from nano Fe_2O_3 to MWCNT. This increase in hardness is originated from the physical and chemical nature of nanoparticles and their compatibility with the ABS polymer matrix. Also Figure 3 reveals that hardness increases by increasing the rotational speed from 500 to 800 rpm and by more increase of the rotational speed from 800 to 1250 rpm, the value of hardness decreases. This reduction of hardness value is because of the high temperature caused by excessive friction between tool and polymer matrix.

This temperature increase causes ABS polymer to soften and consequently directs the flow of molten materials to sides and leads to the formation of a lake of materials in the section of the process. Therefore the second level of the rotational speed parameter was selected as the optimal level.

(a)



(b)



(c)

Figure 2. Nanocomposite samples produced using three different nanoparticles, (a) nanoclay, (b) Fe_2O_3 and (c) MWCNT

TABLE 4. Results of hardness test of FSP polymeric specimens

Sample no.	Shore-D hardness
1	70.66
2	72.78
3	76.68
4	75.02
5	77.06
6	71.76
7	76.96
8	77.60
9	76.14
Pure sample	68.17

Also, it was found that by implementing higher transverse speeds an increase in the hardness can be observed. This increase in hardness can be due to the fast movement of the tool and faster cooling speed of materials in high transverse speed. In other words, in low transverse speeds, the tool moves with a lower speed through the materials and consequently, the heat originated from friction and fluidity of polymer matrix is increased. Therefore, the materials flow to the sides and due to lack of materials in the section of the process, the hardness of FSP polymeric specimens decreases. The third level of transverse speed was selected as the optimum level.

The results of S/N ratio analysis are given in Table 5. According to Table 5, transverse speed has the most significant influence on the hardness value of FSP polymeric specimens. The next factor is the nanoparticle type as reinforcement which is the next most effective parameter with a low difference from first parameter. Also, the rotational speed is determined as a parameter that has the least effect on the hardness value of FSP polymeric specimens. Also, Figure 4 indicates the effect of the interaction of parameters on the results of hardness test.

Figure 4 illustrates that if the transverse speed is set to the third level (63 mm/min), the variations of the two other parameters did not have a significant effect on the hardness of FSP samples.

TABLE 5. Results of S/N ratio analysis on hardness of FSP polymeric samples

Level	Nanoparticle type	Rotational speed (rpm)	Transverse speed (mm/min)
1	37.31	37.40	37.30
2	37.45	37.59	37.46
3	37.72	37.48	37.72
Delta	0.41	0.19	0.42
Rank	2	3	1

Also, samples containing carbon nanotubes have the highest values of hardness under different conditions compared to other nanoparticles.

Therefore, the third level of nano particle type (carbon nanotube), the second level of rotational speed (800 rpm), and third levels of transverse speed (63 mm/min) were selected as the optimum levels to achieve maximum hardness in FSP polymeric samples. Because no sample was produced using these levels of parameters due to Table 2, the optimal sample was produced and its hardness was measured. The Shore D hardness of optimum FSP sample was 79.72 that indicates the hardness of the specimens was improved by approximately 17% compared to a pure sample.

4. 2. SEM Images FSP sections of the ABS sheets were analyzed using SEM surface images. Before SEM test, in order to generate conductivity on the surfaces of polymeric samples, their surfaces were coated with Au. Figures 5, 6 and 7 illustrate the SEM images of different FSP specimens in this study. Three different nanoparticles were used. The distribution of nanoparticles in outer layers of the polymer sheets can be observed using these images. Occurrence of agglomeration causes nonuniform distribution which is not desirable.

Figure 5 shows the SEM pictures of nanocomposite samples containing nanoclay particles at different processing conditions; whiles Figures 6 and 7 indicate the nano Fe_2O_3 and MWCNT ones, respectively.

As it can be observed in the SEM pictures, regardless of the type of nanoparticles, when the rotational speed is set to the second level (800 rpm) nanoparticles are dispersed appropriately in the polymer matrix due to good melting and mixing with the polymer matrix (samples 2, 5 and 8). Although the results illustrate that almost all nanoparticles were appropriately dispersed in the polymeric matrix especially at low concentrations.

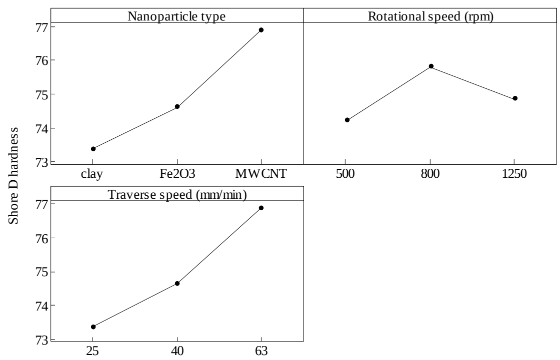


Figure 3. Main effect of parameters on hardness of FSP polymeric samples

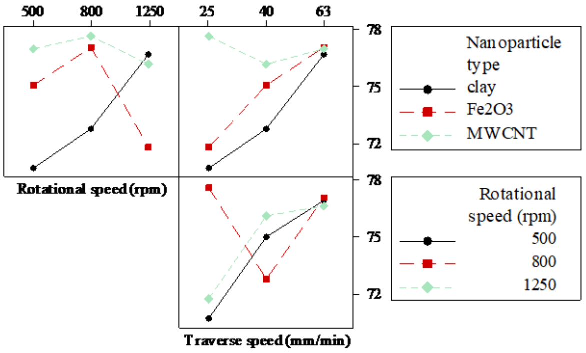


Figure 4. Interaction of parameters on the hardness of FSP polymeric samples

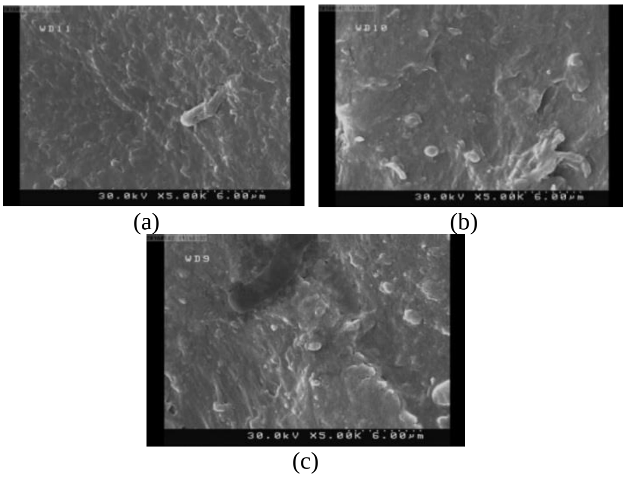


Figure 5. The SEM images of samples containing nanoclay (a) Sample 1, (b) Sample 2 and (c) Sample 3

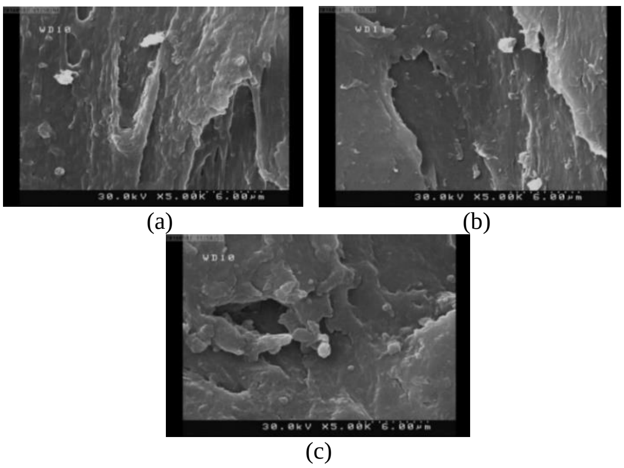


Figure 6. The SEM images of samples containing nano Fe_2O_3, (a) Sample 4, (b) Sample 5 and (c) Sample 6

The best dispersion of nano particles belongs to nano Fe_2O_3 in sample 5 with the rotational speed of 800 rpm and transverse speed of 63 mm/min.

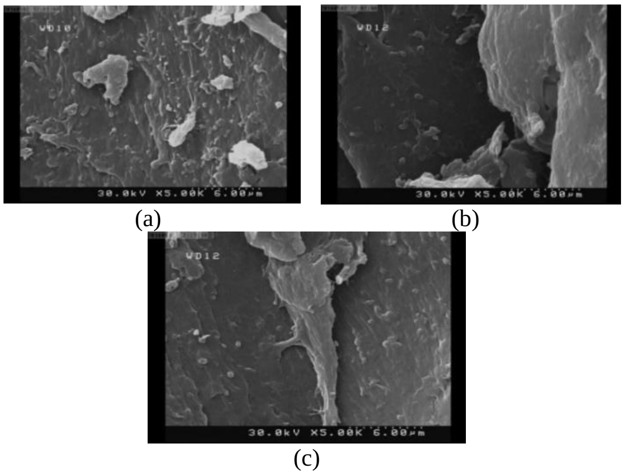


Figure 7. The SEM images of samples containing MWCNT, (a) Sample 7, (b) Sample 8 and (c) Sample 9

It was also concluded in the previous section that the highest hardness is also obtained when carbon nanotubes were used and the rotational speed and transverse speeds are set to 800 rpm and 63 mm/min, respectively. Therefore, it seems that the processing conditions are the most effective factors on the distribution of nanoparticles in the polymer matrix.

5. CONCLUSIONS

In the present study, the effect of material parameters (nano particle type) and process parameters (rotational and transverse speed of tool) on the hardness and morphological properties of FSP sections of ABS polymer was investigated. Hardness and SEM tests were carried out on the injection molded and friction stir processed samples. The results indicated that the addition of carbon nanotubes caused a significant increase in the hardness value of FSP polymeric samples. Also, transverse speed and nanoparticle type were introduced as the most effective parameters on the hardness of FSP specimens. The hardness of FSP specimens was improved approximately by 17% compared to a pure sample. Results of SEM test show that regardless of nanoparticle type when the rotational speed is set to the optimal level, nanoparticles were dispersed appropriately in the ABS polymer due to the better melting and mixing with the polymer matrix.

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چکیده

در پژوهش حاضر، اثر افزودن نانو ذرات رس، اکسید آهن و نانو لوله‌های کربنی چند دیواره به مقطع فرآیند اصطکاکی-اغتشاشی در سطح پلیمر اکریلونیتریل بوتادین استایرن (ABS) و نیز شرایط مختلف فرآیندی بر روی سختی و ساختار نانوکامپوزیت حاصل مورد مطالعه قرار گرفته است. بدین منظور، ابتدا صفحات پلیمری ABS در ابعاد مناسب به روش قالبگیری تزریقی تولید شد. سپس شیاری با عمق و عرض متناسب با ابعاد ابزار فرآیند اصطکاکی-اغتشاشی در سطح ورق‌های ABS با استفاده از دستگاه فرز افقی ایجاد گردید. در ادامه نوع نانوذره، سرعت دورانی و سرعت پیشروی ابزار فرآیند اصطکاکی-اغتشاشی (هر کدام در سه سطح مختلف) به عنوان پارامترهای کنترلی آزمایش‌ها انتخاب شد و طراحی آزمایش مناسب، مطابق با آرایه متعامد L_9 روش تاگوچی انجام گردید. سپس سه نوع نانو ذرهٔ ذکر شده به شیارها اضافه شد و نمونه‌ها در شرایط مختلف آزمایش‌ها، با استفاده از ابزار ساده استوانه‌ای در دستگاه فرز عمودی تحت فرآیند اصطکاکی-اغتشاشی قرار گرفتند. در ادامه مقاطع حاصل از فرآیند اصطکاکی-اغتشاشی تحت آزمایش سختی سنجی قرار گرفت. مشخص گردید که افزودن نانو ذرات موجب افزایش سختی سطح پلیمر شده است. همچنین بیشترین سختی در سطح نمونه‌هایی که از نانو لوله کربنی چند دیواره به عنوان تقویت‌کننده استفاده شده بود، مشاهده گردید. سپس مقاطع فرآیند اصطکاکی-اغتشاشی نمونه‌ها تحت تصویربرداری میکروسکوپ الکترونی روبشی قرار گرفت. تصاویر حاصل از سطح مقاطع نمونه‌ها نشان داد که شرایط فرآیندی تاثیر قابل توجهی در پخش ذرات نانو در ماتریس پلیمری داشته است.

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