



Welding Properties of Polymeric Nanocomposite Parts Containing Alumina Nanoparticles in Friction Stir Welding Process

R. Hasanzadeh^{*a}, T. Azdast^a, A. Doniavi^a, S. Babazadeh^a, R. E. Lee^b, M. Daryadel^a, S. M. Shishavan^b

^a Mechanical Engineering Department, Urmia University, Urmia, Iran

^b Microcellular Plastics Manufacturing Laboratory, Department of Mechanical and Industrial Engineering, University of Toronto, Toronto, Canada

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ABSTRACT

Although in recent years, welding of polymers has been developed but welding of polycarbonates is still faced with serious challenges such as improving the quality of welded section. In the present study, mechanical properties of polycarbonate friction stir welded samples with different nano alumina content were investigated. For this purpose, firstly polycarbonate (as matrix) was melt compounded with nano alumina in variant weight percentages including 0, 1, 2 and 3% using a twin-screw extruder. Then, nanocomposite samples were produced using an injection molding machine and were friction stir welded with a special tool on a milling machine. The effects of weight percentage of nano alumina, travel and rotational speeds (all in four levels) were investigated on the tensile strength and hardness of the welded nanocomposite samples according to a L_{16} orthogonal array of Taguchi method. According to the obtained results, the weight percentage of nano alumina is the most effective parameter on the tensile strength and hardness of welded nanocomposite specimens. By increasing the percentage of nano alumina to 1%, tensile strength increased. However, by increasing the nano alumina more than 1%, this strength reduced due to agglomeration of nanoalumina in high weight percentages. Results also demonstrated that processing parameters do not affect the mechanical properties of welded nanocomposite samples significantly.

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1. INTRODUCTION

Friction stir welding (FSW) was invented as a solid state joining technique and was initially applied to aluminum alloys. In FSW process a non-consumable rotational tool with a specially designed pin and shoulder is inserted into the abutting edges of sheets to be joined and subsequently traversed along the joint line. The generated heat in this process is due to the friction between interfaces and large plastic deformation. This generated heat causes a plasticized zone to form around the tool. The rotating tool moves along the joint line and subsequently, a consolidated solid phase joint is formed [1-3].

In most engineering applications, a combination of materials properties was needed; such as high strength, low weight and good abrasion resistance, thus researchers have developed a new class of materials named composites [4]. Composites have been formed from at least two components with different physical and chemical properties. Nanocomposites have special significance in the nanotechnology; therefore they attracted many attentions in both scientific and industrial aspects in recent years [5]. In nanocomposites, at least one of the components has at least one dimension in the nanometer range (between 1 to 100 nm). In recent years, nanocomposites have been significant improvements because of change in materials structure at the nanometer scale and improve properties of composites [6].

Polycarbonate (PC) is one of the important thermoplastics and has high impact resistance,

*Corresponding Author's Email: re.hasanzadeh@urmia.ac.ir (R. Hasanzadeh)

transparence, high temperature resistance and dimensional stability. PC is one of the toughest transparence materials and has low cost and weight and it is widely used in automotive, medical, computer and food packaging.

The design of experiments (DOE) is an important statistical method in engineering sciences. DOE investigates impact of effective parameters on outputs by reducing the number of tests, time and cost of experiments. Taguchi method is one of the most widely used methods in DOE. Taguchi method is a combination of statistical and mathematical techniques used in experimental studies. This method can determine the optimum conditions with the minimum number of experiments [7]. Taguchi method introduces a loss function that is presented as signal to noise ratio (S/N). Among these ratios, three ratios have been proposed as the standard ratios that are widely used in investigations. These ratios is expressed in Equation (1) to (3) that are used to determine S/N ratio for nominal is best (reduce variability around the target value), larger is better (achieve optimal with growing response rate) and smaller better state (achieve optimal with the small response rate), respectively. In these equations, y is response variable, s is standard deviation and n is the number of repeat experiments. For each parameter, regardless of the type problem, the optimal level is the level with the highest S/N ratio [8].

$$S/N=10\log\left[\frac{\bar{y}^2}{s^2}\right] \quad (1)$$

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

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

There are many researches in the field of welding of polymers and polymeric nanocomposites. Sorensen et al. [9] investigated the effects of tool rotational and travel speeds and kind of welding tools on the tensile and torsional strengths and impact resistance of acrylonitrile butadiene styrene (ABS) sheets. Results showed that rotational speed had the greatest effect on the increasing of tensile strength and decrease of the impact resistance. Also, increasing of the travel speed was lead to decrease of tensile strength and increasing of impact resistance. Both of rotational and travel speeds had a little effect on the torsional modulus. Also kind of welding tools had not a significant effect on the properties. Strand et al. [10] investigated the effects of FSW processing parameters on the properties of polypropylene (PP) sheets by hot shoe method. They found that for high tensile and torsional strengths and uniform microstructure, shoe temperature, pressure time and pin diameter should increase and the travel speed decrease. The effects of rotational and welding speeds, pin diameter and hold time in FSW of high density

polyethylene (HDPE) sheets using Taguchi method were investigated by Rezgui et al. [11]. The results showed that the maximum temperature is increased by increasing rotational and welding speeds and pin diameter. In addition, by increasing rotational speed and pin diameter, maximum stress increased. The results also demonstrated that processing parameters had not significant effect on the yield strength. Bagheri et al. [12] investigated the effects of rotational and travel speeds and shoe temperature in three levels on mechanical properties of welding of ABS sheets. They concluded that by increasing rotational speed and shoe temperature, tensile strength increases but by increasing travel speed this strength reduces.

In this research work, the effect of different parameters on mechanical properties of friction stir welded polycarbonate (PC) sheets is investigated. For this purpose different amount of nanoalumina is added to PC. Furthermore, FSW parameters including rotational and travels speeds are considered in different levels. The optimized conditions are studied using Taguchi approach and optimum levels of considered parameters in terms of maximum mechanical properties (i.e. tensile strength and hardness) of welded zone are obtained. The contribution of parameters on properties of welded zone is also investigated using signal to noise ratio and ANOVA analysis of Taguchi method.

2. EXPERIMENTAL

2. 1. Materials and Equipment

In this study, polycarbonate (PC) with Trade name of Hopelex PC-1100U with melt flow index (MFI) of 10g/10min (300°C, 1.2kg) is used as the polymeric matrix. Gamma nano alumina (manufactured by US Research Co., USA) with density of 3.96 cc/g, average grain size of 20 nm and purity of 99% is used as the reinforcement. Figure 1 shows the SEM image of the supplied alumina nanoparticle.

A ZSK-25 twin-screw extruder with 10kg/h extruding capacity is used for melt compounding of the materials and preparation of nanocomposites with different weight percentages of nanoparticle. An NBM HXF-128 injection molding machine is used for injection molding of specimens. A mold of injection molding machine with dimension of 17.5*8 cm² and thickness of 3.6 mm is used in experimentations (Figure 2). Schematic of the pin (made of steel VCN150) with height and diameter of 3 and 5 mm and shoulder diameter of 10 mm respectively, is shown in Figure 3. FP4M milling machine with four degrees of freedom has been used for welding of nanocomposite specimens. The NCC9012 laser cutting machine is used for cutting of welded nanocomposite samples. A Gotech-AI-7000M tensile test machine is used to calculate tensile strength of welded nanocomposite samples with maximum

capacity of 200 KN and 10 mm/min speed of the test. Indentec universal hardness test machine is used for Rockwell hardness test with accuracy of 0.1.

2. 2. Design of Experiments In this study, tensile strength and Rockwell hardness of friction stir welded polycarbonate nanocomposite samples as the response variables have been studied. The effects of weight percentage of nano alumina, rotational and travel speeds in four levels have been investigated. Therefore, nano alumina was added in four levels i.e. 0, 1, 2 and 3 wt% for investigating the effect of addition of nano alumina on the mechanical properties of FSW and comparison with the pure PC samples. Levels of FSW parameters are selected according to the available levels of milling machine and experimental observations. For example the maximum rotational speed of milling machine was 2500 rpm.

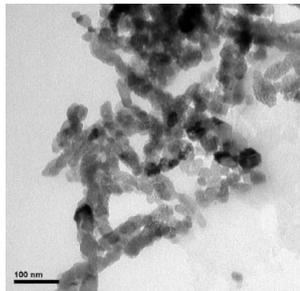


Figure 1. The SEM image of alumina nanoparticles

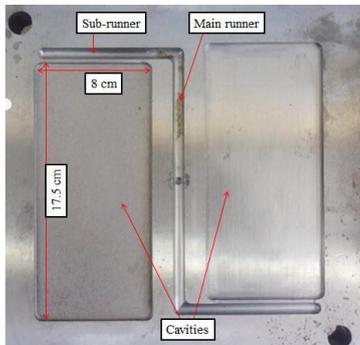


Figure 2. The used mold of this study

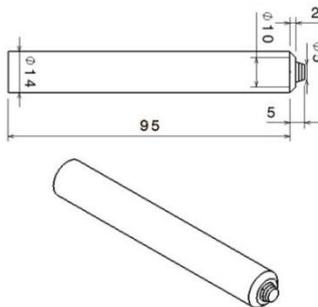


Figure 3. The schematic of pin used for friction stir welding in this research

On the other hand, experimental results show that with rotational speeds smaller than 1250 rpm, since the generated heat was not high enough, the resulted welds were not acceptable. Therefore, 1250 and 2500 rpm were considered as the minimum and maximum rotational speeds, respectively and two other levels (1600 and 2000 rpm) were selected in between with the same distance.

It was also observed that when travel speed is less than 8 mm/min, contact time of rotational tool with nanocomposite samples surfaces is long and the generated heat is too much. Similarly, in the travel speed more than 20 mm/min, due to the fast movement of tool, deformations and voids are observed in weld line of welded samples, which results in a poor and undesirable weld quality (Figure 4). Therefore, travel speeds were selected in levels of 8 and 20 mm/min and two levels in between (i.e. 12 and 16 mm/min).

According to processing parameters and their levels, a L_{16} orthogonal array of Taguchi method is selected for design of experiments using Minitab software [13]. This array of experiments is shown in Table 1.

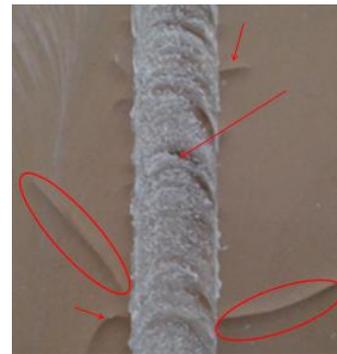


Figure 4. The inappropriate welded sheets in 24 mm/min travel speed

TABLE 1. Design of experiments according to L_{16} orthogonal array of Taguchi method

Trial no.	Nano alumina (wt%)	Travel speed (mm/min)	Rotational speed (rpm)
1	0	8	1250
2	0	12	1600
3	0	16	2000
4	0	20	2500
5	1	8	1600
6	1	12	1250
7	1	16	2500
8	1	20	2000
9	2	8	2000
10	2	12	2500
11	2	16	1250
12	2	20	1600
13	3	8	2500
14	3	12	2000
15	3	16	1600
16	3	20	1250

2. 3. Preparation of Specimens Before melt compounding, raw materials are dried using an oven at 80 °C for 2 hours. Then, PC and nano alumina are extruded in a twin-screw extruder with different weight percentages of nano alumina at melt temperature of 240 °C and screw speed of 250 rpm. Prior to injection, the obtained nanocomposite granules are dried using the dryer unit of injection molding machine at 80 °C for 24 hours. Since the cooling of extruded material has been performed in a water tank this stage of drying needs longer time in comparison with the first step of drying (i.e. drying prior to extrusion) [14].

According to the processing conditions of Table 2, specimens were produced using injection molding machine. Then the injected nanocomposite plates were fixed for welding on a milling machine using special fixture and clamps. Parallel fixtures with moving of tool were set by Indicator clock with accuracy of 0.01. The procedure of sheets welding is shown in Figure 5. Some welded nanocomposite samples are also shown in Figure 6. Later on, specimens were cut for characterization from the welded plates using a laser cutting machine.

Tensile and hardness tests were conducted to investigate mechanical properties of welded nanocomposite samples. In order to increase the accuracy of tensile test, at least three specimens were selected from each sample and the average quantity was reported as the final strength of sample based on ASTM D638 standard [15].

TABLE 2. Injection molding processing conditions

Parameter	Adjusted value
Injection temperature (°C)	310
Injection pressure (MPa)	110
Hold pressure (MPa)	90
Cooling time (s)	20
Holding pressure time (s)	3
Mold temperature (°C)	100



Figure 5. Fixture, clamps and specimens mounted on the milling machines

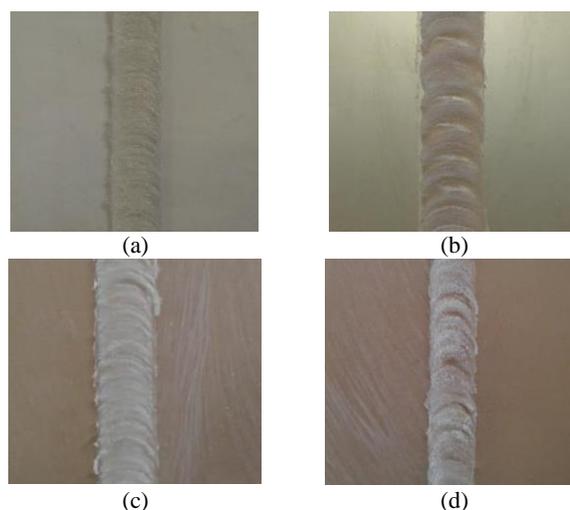


Figure 6. The welded nanocomposite samples: a) Trial 3, pure polycarbonate, b) Trial 5 containing 1 wt% nano alumina, c) Trial 11 containing 2 wt% nano alumina and d) Trial 15 containing 3 wt% nano alumina

Hardness test of welded nanocomposite samples was also performed based on Rockwell-L method. For this purpose, specimens were tested at least in five points and the average quantity was reported as the final hardness of welded nanocomposite samples.

3. RESULTS AND DISCUSSION

Results of the tensile and hardness tests are shown in Table 3. Main goal of this research is maximization of tensile strength and hardness, therefore S/N ratio analysis has been used in larger is better state (Equation 2).

TABLE 3. Results of tensile and hardness tests of friction stir welded nanocomposite samples

Trial no.	Tensile strength (MPa)	Rockwell hardness
1	67.87	76.43
2	64.32	70.83
3	117.17	68.30
4	87.62	63.67
5	105.35	65.50
6	117.28	55.50
7	91.57	74.67
8	93.12	60.93
9	79.13	56.70
10	80.03	66.43
11	84.06	55.90
12	63.04	53.30
13	63.49	67.53
14	58.11	58.60
15	64.91	67.63
16	73.09	60.67

Figure 7 shows the welded nanocomposite samples of this research after performing the tensile test according to mentioned standard. As an example, the stress-strain curve of the sample no. 6 which is containing 1 wt% alumina nanoparticles is shown in Figure 8.

3. 1. Tensile Strength

Signal to noise ratio diagram of Taguchi design for tensile strength of this research is shown in Figure 9. Maximizing the S/N ratio offers better performance and shows the optimum condition. Consequently, according to the S/N ratio results of Figure 9 it could be concluded that the tensile strength is optimum when weight percentage of nano alumina, travel and rotational speeds are in levels of 2, 3 and 3, respectively. Moreover, the maximum difference of S/N ratio for each parameter shows the ranking of importance of parameters. In other word, results of Figure 9 can also depict that weight percentage of nano alumina, rotational and travel speeds are the most important effective parameters on tensile strength of welded nanocomposite samples, respectively.

The results demonstrated that tensile strength increases by increasing the weight percentage of nano alumina to 1%. This could be due to the special properties of nano alumina (such as high mechanical properties) transfer to polymer. However, when nano alumina is added more than 1%, the tensile strength will be decreased due to the agglomeration of the nano alumina in high weight percentages follow by stress concentration [16]. Figure 10 shows the agglomeration of nanoalumina in PC in high weight percentage content.

Regarding the welding processing parameters, when the travel speed is small, due to the long contact time of rotational tool with the surfaces nanocomposite samples, the amount of generated heat will be large. This large amount of the heat leads to a decrease in the tensile strength. As a result if the travel speed is increased to 16 mm/min, the tensile strength property will be improved. However, according to S/N ratio diagram, by more increasing of the travel speed, the tensile strength decreases due to the fast movement of tool and consequently low heat generation which is not desirable for FSW process. Furthermore the S/N ratio diagram indicates that rotational speed does not have considerable effect on the tensile strength of welded nanocomposite samples.

Another result obtained from Taguchi approach is that the predicted optimum condition (i.e. 1wt% of nano alumina, 16 mm/min of travel speed and 2000 rpm of rotational speed) will results in a tensile strength of 118.37 MPa. The S/N ratio diagram shows ranking of the importance of parameters on the response variables as well. For this purpose, the analysis of variance (ANOVA) for tensile strength has been performed. For that firstly normal probability of tensile strength is

investigated (Figure 11). According to the normal probability diagram, P-value is more than 0.05 (that has been determined as the amount of error by Minitab software), which means data of the tensile strength tests have a normal distribution.

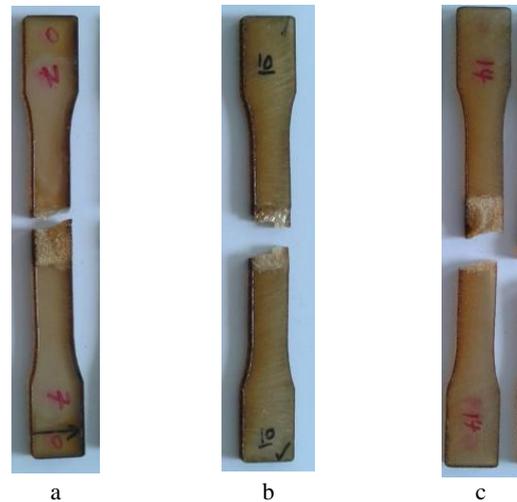


Figure 7. Welded nanocomposite samples after performing the tensile test containing a) 1 wt%, b) 2 wt% and c) 3 wt% of alumina nanoparticles

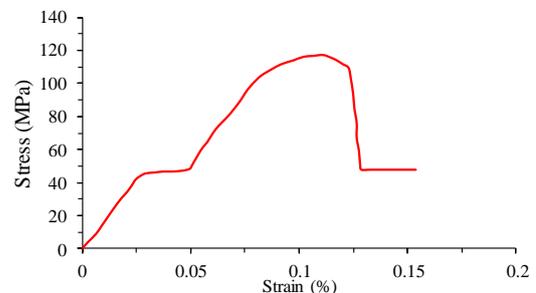


Figure 8. Stress-strain curve of sample no. 6 containing 1 wt% alumina nanoparticles

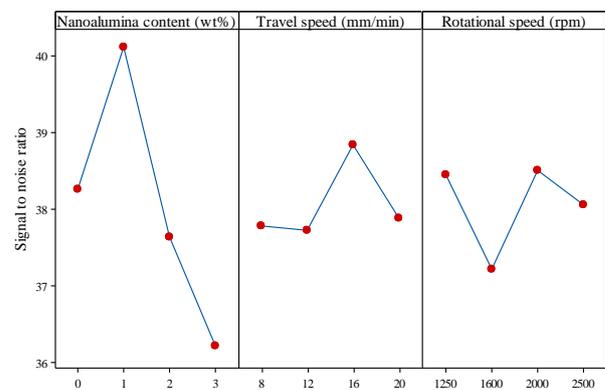


Figure 9. Signal to noise ratio diagram of tensile strength

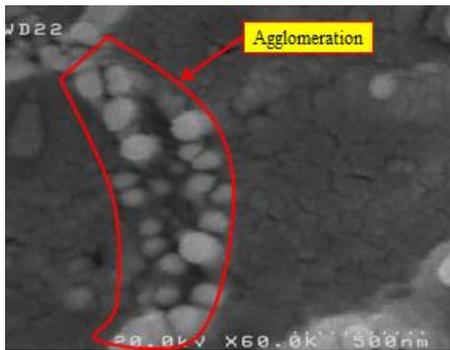


Figure 10. The observed agglomeration of alumina nanoparticles in PC matrix

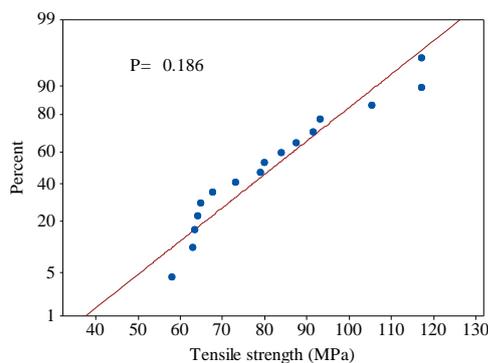


Figure 11. Normal probability diagram of tensile strength

Analysis of variance of tensile strength is indicated in Table 4. According to analysis of variance, the weight percentage of nano alumina is the most effective parameter on the tensile strength with 63.9% of contribution. Rotational and travel speeds are next effective parameters, respectively. Besides according to these results, the error has 20.8% of contribution on the tensile strength which could refer to the effects of some processing parameters that were assumed to be constant and their effect has been neglected.

Another interesting result of the ANOVA is the interaction effect of parameters. Interaction effect of weight percentage of nano alumina and rotational speed that have more influence on the tensile strength of welded nanocomposite samples is illustrated in Figure 12. The results show that the tensile strength of welded nanocomposite samples decreases almost linearly by increasing of rotational speed for 1 wt% of nano alumina. It could also see from the Figure 12 that in low rotational speeds (levels 1 and 2), the tensile strength of welded nanocomposite samples containing 1 wt% of nano alumina is dramatically high compare to other conditions.

3. 2. Rockwell Hardness Following is the study of the effect of processing parameters on the Rockwell hardness of welded nanocomposite samples. S/N ratio

diagram has been used in larger is better state which is shown in Figure 13. According to S/N ratio diagram, the hardness of welded samples is maximum when the polycarbonate is pure, rotational and travel speeds are 2500 rpm and 16 mm/min, respectively. In other words, to achieve the optimum conditions for more hardness of welded samples, the polycarbonate should be pure and the rotational and travel speeds should be in levels of 4 and 3, respectively. The Rockwell hardness of this optimum condition is obtained 76.68 using Taguchi method prediction. Regarding the ranking of the effective parameters, weight percentage of nano alumina, rotational and travel speeds are the most important parameters on the hardness of welded nanocomposite samples, respectively.

Results of Figure 13 shows that increasing the weight percentage of nanoalumina to 2 wt% caused to a decrease in hardness of samples. This could be due to the fact that rotational movement of tool will orient the nano alumina particles towards the core. Therefore, there will be a lack of the nano alumina particles in the surfaces of the samples; consequently the hardness will be decreased. However, the hardness will increase with the nano alumina particles content more than 2 wt %, probably due to the existence of some nano alumina particles in the surfaces of samples. The results also demonstrate that increasing rotational speed up to level of three has not considerable effect on the hardness of welded nanocomposite samples.

TABLE 4. Analysis of variance of tensile strength

Variable	Contribution (%)
Nanoalumina content (wt%)	63.9
Travel Speed (mm/min)	6.8
Rotational speed (rpm)	8.5
Error	20.8
Total	100

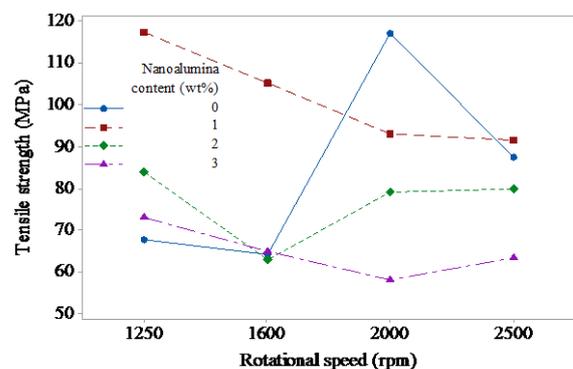


Figure 12. Interaction plot of effect of nano alumina and rotational speed on the tensile strength

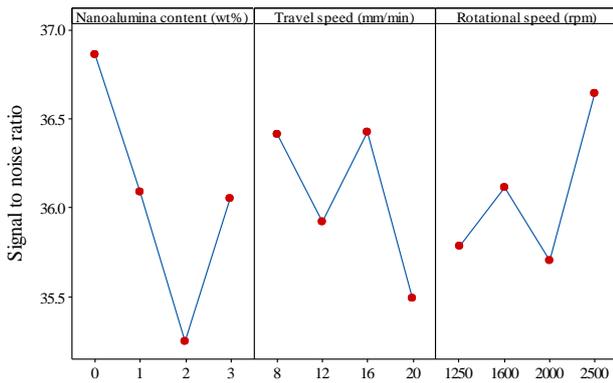


Figure 13. Signal to noise ratio diagram of hardness

However, hardness has been increased in level four due to the high rotational speed of tool follow by orientation of nano alumina towards welding surfaces. Also, travel speed has no justifiable effect on the hardness of welded nanocomposite samples.

Normal probability diagram of the hardness test presented in Figure 14 shows that P value is more than 0.05 (amount of error), which means data of hardness of welded nanocomposite samples have also normal distribution. Analysis of variance for hardness of welded nanocomposite samples has been shown in Table 5. According to analysis of variance, the weight percentage of nano alumina is the most effective parameter on the hardness of the welded nanocomposite samples with 44.22% of contribution. Travel and rotational speeds are next effective parameters, respectively. Moreover according to the results, the error has 15.95% of contribution on the hardness. Interaction effect of weight percentages of nano alumina and travel speed that have more influence on the hardness of welded nanocomposite samples are depicted in Figure 15. The results indicate that the hardness of welded samples is maximum when the polycarbonate is pure and the travel speed is low. In addition, the hardness of welded samples decreases in pure specimens by increasing the travel speed.

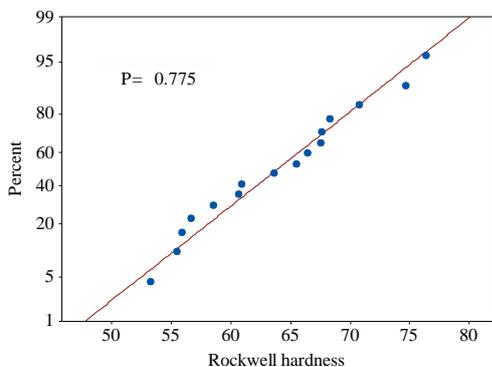


Figure 14. Normal probability diagram of hardness

TABLE 5. Analysis of variance of hardness

Variable	Contribution (%)
Nanoalumina content (wt%)	44.22
Travel Speed (mm/min)	21.60
Rotational speed (rpm)	18.23
Error	15.95
Total	100

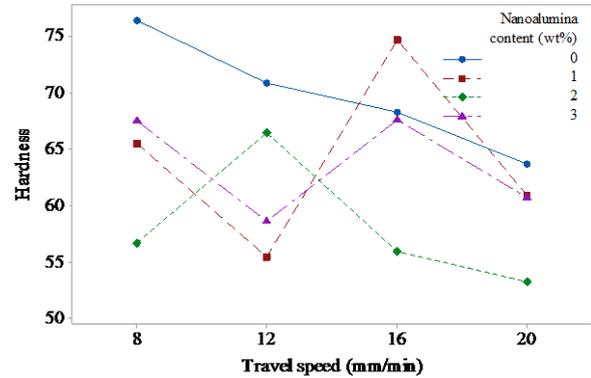


Figure 15. Interaction plot of effect of nano alumina and travel speed on the hardness

Another interesting result is that the hardness of samples containing 1 wt% of nano alumina is maximum and minimum in travel speeds of 16 and 12 mm/min, respectively. At all levels of travel speed except 12 mm/min, the hardness of specimens containing 2 wt% of nano alumina is the minimum hardness.

4. CONCLUDING REMARKS

The effects of the weight percentage of nano alumina, rotational and travel speeds were investigated on the tensile strength and hardness of PC/ alumina nanoparticles welded nanocomposite samples. The results demonstrated that the weight percentage of nano alumina is the most effective parameter on the tensile strength and hardness of welded nanocomposite samples. According to S/N ratio diagram of Taguchi approach, the optimum conditions for the tensile strength of welded samples were: 1 wt% of nano alumina, travel speed of 16 mm/min and rotational speed of 2000 rpm. The results also revealed that the hardness of welded samples is optimum in conditions of 0 wt% of nano alumina, rotational speed of 2500 rpm and travel speed of 16 mm/min. Moreover, the results indicated that friction stir welding processing parameters do not have a significant effect on mechanical properties of welded nanocomposite samples.

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^a Mechanical Engineering Department, Urmia University, Urmia, Iran

^b Microcellular Plastics Manufacturing Laboratory, Department of Mechanical and Industrial Engineering, University of Toronto, Toronto, Canada

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Nanoalumina

Taguchi method

اگرچه در سالیان اخیر جوشکاری پلیمرها توسعه یافته است ولی جوشکاری پلی کربنات‌ها هنوز با چالش‌های جدی از جمله بهبود کیفیت مقطع جوش روبه رو است. در پژوهش حاضر، خواص مکانیکی نانوکامپوزیت پلی کربنات جوشکاری شده به روش اصطکاکی اغتشاشی با افزودن مقادیر مختلف ذرات نانوالومینا مورد بررسی قرار گرفت. ذرات نانوالومینا در درصدهای وزنی ۰، ۱، ۲ و ۳ توسط دستگاه اکسترودر دوماردونه به پلی کربنات که به عنوان ماده پایه می‌باشد، به روش اختلاط ذوبی افزوده گردید و نمونه‌های نانوکامپوزیتی تزریق شدند. نمونه‌های تولید شده دو به دو به صورت صحیح روی دستگاه فرز بسته شده و با استفاده از ابزار مخصوص به روش اصطکاکی اغتشاشی جوش شدند. تأثیر درصد وزنی نانوالومینا، سرعت پیشروی و سرعت دورانی (هرکدام در چهار سطح) بر روی استحکام کششی و سختی محل جوش نمونه‌های جوشکاری شده بر اساس طراحی آزمایش تاگوچی مطابق آرایه متعامد L_{16} بررسی شد. نتایج نشان داد که درصد وزنی نانوالومینا تأثیرگذارترین پارامتر روی استحکام کششی و سختی نمونه‌های نانوکامپوزیتی جوشکاری شده می‌باشد. به طوری که با افزایش درصد وزنی نانوالومینا تا ۱ درصد، استحکام کششی نمونه‌ها افزایش می‌یابد اما با افزایش بیشتر درصد وزنی نانوالومینا، این استحکام به دلیل کلوخه‌ای شدن ذرات نانوالومینا در درصدهای بالا، کاهش می‌یابد. نتایج همچنین بیانگر این موضوع بودند که تغییرات شرایط فرآیندی جوشکاری، تأثیر قابل توجهی روی خواص مکانیکی نمونه‌های نانوکامپوزیتی نداشتند.

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