

EFFECT OF ADDING BORON CARBIDE (B_4C) TO POLYMER FOR PRODUCING SURFACE COMPOSITE BY FRICTION STIR PROCESSING

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ABSTRACT

Friction stir processing (FSP) is a new solid state technique, it is employed for the improvement of the mechanical properties of a material and the production of surface layer composites instead of conventional processing technologies. This research aims to study the ability of applying Friction Stir Processing (FSP) to modify the surface of high density polyethylene (HDPE) reinforcing by B_4C with a particle size of $0.4\mu m$, Groove in the middle of HDPE surface made to fill by B_4C . Variety in the groove depth (0.6, 1.2 and 1.8)mm used according to B_4C ratio on HDPE substrate particles. Friction stir process was carried out, using tool with cylindrical shape of pin and shoe tool to produce surface layer composite. The effect of processing parameters including rotational and transverse speeds on the mechanical properties of composite layer was studied. Wear test results show a pronounced improvement in wear resistance of HDPE surface through reinforcement additions of B_4C at a ratio (5%, 10% and 15%), where wear rate improved by (60%, 71% and 63%) respectively, as compared with as received HDPE, the surface composite HDPE/ B_4C have good wear resistance. Hardness test results indicate that the hardness of composite layer reinforced with (5%,10% and 15%) particles improved by (26%, 35% and 28%) respectively as compared with received HDPE. OM revealed that high tool rotational speed resulted in homogeneous distribution of B_4C particles and vice versa.

Keyword: Friction stir process, Composite Materials, Wear Resistance .

تأثير اضافة كاربيد البورون الى المادة الراتنجية لانتاج مركبات سطحية باستخدام طريقة

الخلط بالاحتكاك

بشائر علي حبيب احمد عودة جاسم

الخلاصة:

عملية الخلط بالاحتكاك هي تقنية جديدة لتصنيع المواد في حاله الصلبه، وهي تستخدم لتحسين الخواص الميكانيكية للمادة وإنتاج مركبات الطبقة السطحية بدلا من تقنيات المعالجة التقليدية. يهدف البحث الى دراسته تطبيق الخلط الاحتكاكي على البوليمر لتكوين مركبات على الطبقة السطحية، وقد تم استخدام (البولي إيثيلين عالية الكثافة) حيث تم تقيبه السطح بواسطة B_4C مع حجم الجسيمات $0.4\mu m$ ، الشق في وسط سطح العينه ثم يملئ من قبل B_4C . التباين في عمق الأخدود (0.6، 1.2 و 1.8) المستخدمة وفقا لنسبة B_4C المستخدم وقد أجريت عملية الاحتكاك الدوراني بها، وذلك باستخدام أداة مع شكل أسطواناني من دبوس و (shoe tool) لإنتاج طبقة سطح مركب. تم دراسة تأثير معاملات المعالجة بما في ذلك السرعات الدورانية والعرضية على الخواص الميكانيكية للطبقة المترابطة. أظهرت نتائج الاختبار تحسنا ملحوظا في مقاومة التآكل لسطح البولي إيثيلين عالي الكثافة من خلال الإضافات التعزيزية ل B_4C بنسبة (5% و 10% و 15%) حيث انخفض معدل التآكل بنسبة (60%، 71% و 63%) على التوالي مقارنة مع كما ورد HDPE، تشير نتائج اختبار الصلابة إلى أن صلابة الطبقة المترابطة المقواة ب5%، 10% و 15% من الجسيمات تحسنت بنسبة (26%، 35% و 28%) وعلى التوالي مقارنة مع HDPE المستقبلية. أن سرعة عالية أداة التناوب أدى إلى توزيع متجانس من جزيئات B_4C والعكس بالعكس.

INTRODUCTION

FSP is a solid-state processing technique depended on friction stir welding basics, which was contained a special design of rotating tool with moves down the surfaces of material plates. This is achieved by generating a highly plastically deformed region by the associated stirring action [P. Asadi et. al, 2011].

The FSP method developed by Mishra et al. It is considered as a common process for microstructural modification. It was based on the traditional FSW, which was studied and used since its discovery in 1991[C. Huang et.al, 2016]. Principles of FSP was similar that friction stir welding and FSP have the same facilities. FSW and FSP depend on the work done by forcing a rotating tool into the workpieces of two materials and followed by moving a rotational tool on the gap between two materials to create the intimate contact at the interface [K. Panneerselvam & K. Lenin ,2013]. Friction stir processing at first time was used as commonly with aluminum, but it was also used for FSW and FSP other alloys, for example, Ni-based intermetallic compound modified bronzes, Zr alloys, Mg alloys and tool steels. It was noted that FSP polymers and polymeric composites are still much less studied than FSP metals and metallic composites There are some concerns related to friction stir processing polymers because the rotation of tool pin and the severe plastic flow in the nugget zone could break macromolecular chains and change the properties of the materials [X. Gan, Yong et.al, 2010]. polymeric materials have low hardness, quickly reached by the friction phenomenon due to the extremely low melting temperature, short time of cementing process and have additionally low warm conductivity[O.Salman et.al, 2006]. Thermoplastics like polypropylene and polyethylene have been processed by FSP. They are used in electronic applications and automotive industry due to their higher strength to weight ratio, excellent processing properties, their good performance and favorable low cost. Nevertheless, their uses are limited because of their low hardness, at room temperature have poor effect resistance; the lower one and weak abrasive properties. The inorganic fillers additives have been a common practice in the plastics manufacturing to enhance the mechanical properties of thermoplastics, such as, stiffness, hardness, toughness, mould shrinkage and heat distortion temperatures. Therefore, so much attention has been paid to improve the mechanical properties of thermoplastic in the lately [R. V. Prasad & P.M. Raghava ,2012]. A. Aric and T. Sinmaz 2005 applied friction stir welding (FSW) using the tool of cylindrical shouldered to join the polyethylene sheet. they studied bending and tensile strength of polyethylene joint. They observed that welded samples were fractured without displaying a yield necking. Arici and Selale 2007 studied the influence of tool title angle about polyethylene friction stir welding (FSW). The deduced that, the welding parameters affected ductile properties and break areas of the welds. the thickness of the welding zone diminished with expanding apparatus tilt point which influences the rigidity. Also, the interface line between weld passes was framed shorter for the higher welding speeds on account of the less impact of the frictional warmth.

In this study, FSP was applied in fabricating Polymer Matrix Composite (PMC) by the reinforcing of high density polyethylene (HDPE) with B₄C particles in three ratio (5%, 10% and 15%). The effect of FSP parameters (rotational and transverse speeds) on the mechanical properties (wear resistance and hardness) and microstructure was studied as compared with as received one.

EXPERIMENTAL DETAILS

A 10 mm thick Polyethylene sheet was obtained from local markets. A piece of this material was inspected by infrared spectroscopy (FTIR) to identify and verify the type of this polymer. Reinforcement materials used for the production of metal matrix composite are ceramic particles of boron carbide (B₄C). The average particle size of B₄C (0.4)µm. The machine

used in this research is milling machine (FU251M) made in Bulgaria by Arsenal Company. The tool has been used in the FSP were manufactured from alloy steel X12, the chemical composition of this alloy is present in the **Table(1)**. The geometry of the tool is cylindrical shape. The pin was cylindrical in dimensions(6mm) diameter, (2.5mm) length while the shoulder of (18 mm) diameter shown in the **Fig.(1)**. and the shoe tool which adding to tool in this study as shown in the **Fig. (2)**. the chemical composition of this alloy is present in the **Table (2)**.

After preparation of the sample and fixing will work groove in the middle of the plate. The groove creates by 3mm cutter of milling machine with (0.6,1.2 and 1.8)mm in depth according to B₄C ratio in the groove. The ratio of B₄C from the area of cross section of FSP layers, that was show in **Fig.(3)**

$$\text{Theoretical volume fraction} = \frac{A_g}{PA_t} \times 100\% \tag{1}$$

$$\text{Actual volume fraction} = \frac{A_g}{A_{sc}} \times 100\% \tag{2}$$

$$\text{Area of surface composite}(A_{sc}) = 2 \times \text{triangle} + 2 \times \text{rectangle} + \text{square} \tag{3}$$

$$= 2 \times (1/2 \times 8.5 \times 2) + 2 \times (8.5 \times 0.5) + 3 \times 3.5$$

$$= 36 \text{ mm}^2$$

$$= 36 \text{ mm}^2$$

$$\text{Area of groove}(A_g) = \text{groove width} \times \text{groove depth} \tag{4}$$

$$\text{Projected area of tool pin}(PA_t) = \text{pin diameter} \times \text{pin length} \tag{5}$$

Where:.

groove width= 3mm

groove depth=h

pin diameter = 3mm

pin length= 3mm

At:.

h=0.6mm → Actual volume fraction = 5%

h=1.2mm → Actual volume fraction = 10%

h=1.8mm → Actual volume fraction = 15%

The groove fully filled with reinforcement particles, then tool and shoe tool which used to close the groove to prevent exit particles from the groove during the process without used pin less for this purpose. After covering the groove the FSP tool is inserted into the sample to perform FSP. At the starting point of the sample the tool must be stay some time (30 second) to obtain the necessary heat to soften the base polymer and then the tool move along the process line. During movement the tool in the sample gets mixed between reinforcement particles and matrix plate to result surface composite. At the end of the process tool pulls of the sample and leave a sample to cool as shown in **Fig(4)** . The wear resistance is tested by wear device (pin on disc) type (MT.4003 version 10.0) The sample testing was cut from the stir zone of the FSP sample with a the FSPed sample with a diameter of 20mm according to ASTM G99–04 standardization [P. J., Blau, & K. G. Budinski, K. G. 1999]. The disc (sample) is a fine surface produce by using the shoe tool that which have large effect on the surface roughness, the pin is a Al₂O₃ ball of 6mm diameter. The disc rotational at a constant sliding speed of 700 rpm and the total sliding distance of 1000 m. The loads are (10N, 15N, 20N ,25N,30N), the sample weighted after (15 min) and raise load for 5N. An electron weighing balance with ±0.0001 accuracy are used to measure the weight loss of the sample. Hardness test was performed on samples which had dimensions of (20x20x5) mm according to ASTM D2240, using the Durometer tester. Macro and micro surface structures were carried out by using of optical microscope to study the homogeneity of the mixture.

RESULTS AND DISCUSSIONS

Wear Results

Wear resistance at 5% B₄C The Wear resistance test was made on both, the as received HDPE and processed samples. It was performed by using a pin on disk arrangement **Fig.(5)**. shows illustrates the relationship between the wear resistance at 5% B₄C and the applied load (10,15,20,25,30)N with different of parameters ; rotational speed (500,800 and 1000) rpm and transvers speed (20,40 and 60)mm/min.

The Wear rate for HDPE/surface composite with 5% B₄C increased as the rotational speed increases. High rotational speed for a polymeric material such as HDPE base tends to produce high heat from frictional that causes high mixing between powder with the polymeric material .At high rotational the temperature of material caused high heat from frictional between the tool and material. This can be attributed to low thermal conductivity of polymeric material, which leads to heat concentration in FSP nugget. the composite showed little increase in Wear rate with load, when reinforced with B₄C particles due to the strong bond that was formed between matrix and particle, while HDPE showed a sudden increase in Wear rate with load, which indicates that the mass loss increases. At (1000 rpm) the wear rate was increase with the increase in the transverse speed, This can be attributed to proper melting of the material hence dilution of B₄C powder can occur at low feed rate which would increase the wear , adequate softening of matrix can be achieved when the transvers was increased.

At (800 rpm) the wear rate was increased as the transverse speed increased. The wear rate was reduce at transvers speed 20mm/min due to the suitable amount of heat generation that cause the substrate to soften which enhances mixing quality so the increase in rotational speed which leads to voids content. The maximum wear resistance for HDPE/surface composite reinforced with 5% B₄C was obtained At lower rotational speed (500 rpm) and higher transverse speed (60 mm/min).

HDPE was reinforced with 10% B₄C particles (high abrasive resistance material) with the same processing parameters compared with wear resistance for the HDPE/surface composite with 5% B₄C. The Wear rate of HDPE/ surface composite with 10% B₄C increased as the rotational speed increased. The maximum wear resistance was obtained at rotational speed was 800 rpm and traveling speed was 20mm/min. The proper melting and good wettability between powder and substrate can be achieved at 20 mm/min.

The high ratio 15% B₄C of particles need to increase in rotational speed for distribution the reinforced particle in a large area in the polymer. At 800 rpm the Wear rate was found to be increased as the transverse speed was increased from 20 to 60 mm/min. Anyway, the wear rate was decreased approximately 63 % at 20 mm/min (reaching $15.2 \times 10^{-4} \text{ cm}^3$ at 30 N load) as shown in **Fig.(5)**. In contrast to metals, polymers do not work. harden because no new obstacles are created when the molecules slide past each other. Heat generated during deformation causes a local increase in temperature, further easing plastic deformation. It is observed from experimental work, that an increasing in the amount of B₄C needs more rotational speed to achieve more uniform and homogeneous of distributed the ceramic particles in HDPE matrix. Maximum Wear rate occurs at 500 rpm at 5% of B₄C, while optimum wear rate accrues at 800 rpm and 20 mm/min, when increasing the ratio of B₄C to 10% because the growing of transvers speed lead to gaps increasing and degradation.

Hardness Results

Hardness was measurement by using Shore-D at a different areas of at HDPE/surface composites (retreating zone (RZ), center zone (CZ), advanced zone (AZ)) as shown in **Fig.(6)**. That have higher hardness than the base HDPE polymer. **Fig.(7)(a, b and c)** . show the effect of rotation speed on the hardness of surface composite reinforced by (5%,10% and 15%) B₄C respectively. the hardness was decreased when the rotation speed increase. When tool rotational speed was increased to 1000 rpm, hardness of composite surface was decreased. The hardness was decreased at low transvers speed at the rotational speed 500 rpm. When tool rotational speed was increased to 1000 rpm the hardness decreased which can be attributed to the breaking up of particles agglomerations and then their uniform distribution. At high rotational speed the same amount of particles packed in the groove was distributed into larger area of softened HDPE. Therefore, the hardness of the surface composite becomes lower at 1000 rpm for the three ratio of B₄C. The low ratio 5% and 10% of B₄C have high hardness at 500 rpm with the increase in transvers speed, but at 800 rpm the high hardness at 20 mm/min transvers speed may be because the particles were agglomeration at small area in the composite surface. The high ratio 15% of B₄C give the high hardness at 800 rpm, the due to a lot of mount from reinforced particles need to high rotational speed for homogenous distribution in the layer of surface composite. At high rotation speed 1000rpm the surface composite have a low hardness at all ratio because HDPE sheet begins to melt, burn and degrade. Furthermore, excessive turbulence of mix will occur, which leads to voids content this agree with [M. K. Hussian, (2016)].

Microstructure observations

The distributions of reinforcement particles B₄C in the HDPE matrix was revealed by using optical microscope 400X. The optimum result of the composite surface at HDPE surface reinforced with10% B₄C at rotational speeds (800) rpm and transverse speed (20) mm/min was show in **Fig.(8)**. When the transverse speed was increased that was mean the time of tool residence was decreased. The agglomerations gradually disappeared when tool rotational speed was decreased. Tool rotational speed has two more functions in addition to heat generating due to friction, stirring the softened materials as well as influencing matrix sample flow behavior across the processed zone. Excellent distribution for B₄C was shown in **Fig.(8)**. At center zone, when rotational speed increased to 800 rpm at 20 mm/min, where the average spacing between B₄C particles increased.

CONCLUSIONS

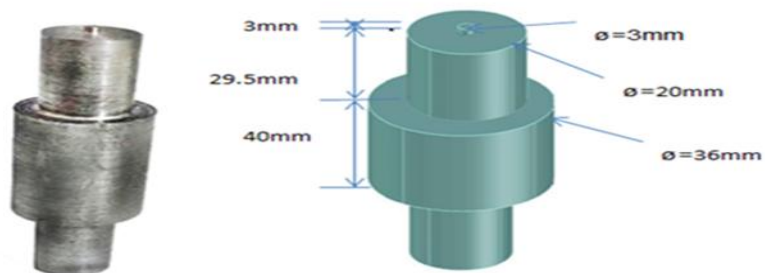
The conclusions derived from this experimental work can be summarized as surface modification of HDPE by continuously stirring B₄C during FSP technique produces layers of composite material with a wide range of B₄C content .The shapes, dimensions, homogeneity and the type of defects composite layers depend greatly on FSP parameters. Low rotational speed causes insufficient melting of the matrix and very high rotational speed leads to high dilution of the particle and matrix which reduces the mechanical properties. The maximum wear resistance and hardness occur at the rotational speed 500 and 800 rpm. The B₄C particles at ratio 10% show excellent wear resistance ,where wear rate decreased by 71% as compared with received HDPE, due to the nature of granules which have high abrasion resistance, compared to 63% and 60% for 15% and 5% Of B₄C ratio respectively. Hardness of composite layer reinforced with B₄C particles at ratio 10% increased 35%higher than that of the as received material compared with 28% and 26% for reinforced with (15% and 5%) B₄C particles ratio respectively compared with received HDPE. The good distribution of particles was accurate at High tool rotational speed and low transvers speed when the ratio was 10% and 15% but at 5% ratio of B₄C the good spreading of particles was accurate at low rotational speed .

Table (1): Nominal Composition of Alloy Steel X12

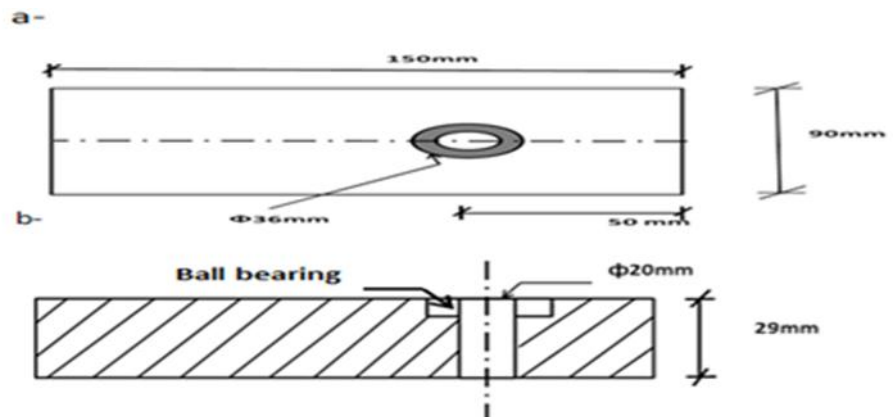
| Element% | Cr% | C% | Si% | Mn% | Fe% |
|--------------|------|------|------|------|-----|
| Actual value | 11.5 | 2.00 | 0.34 | 0.30 | Rem |

Table (2) : Chemical Composition of AA7075 Alloy for shoe tool

| Element | Zn | Mg | Cu | Cr | Mn | Ti | Si | Fe | Al |
|---------|-----|-----|-----|------|-----|-----|-----|-----|------|
| Wt% | 5.6 | 2.5 | 1.6 | 0.23 | 0.3 | 0.2 | 0.4 | 0.5 | rest |



Figure(1): tool



Figure(2): Shoe tool design a . Tope View b. Side View

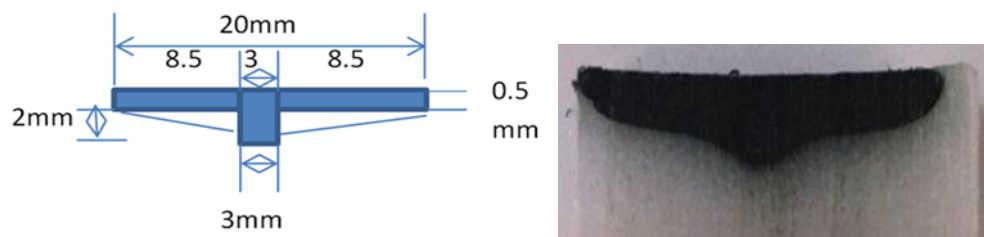


Figure (3): Cross Section of FSP Composite Layer

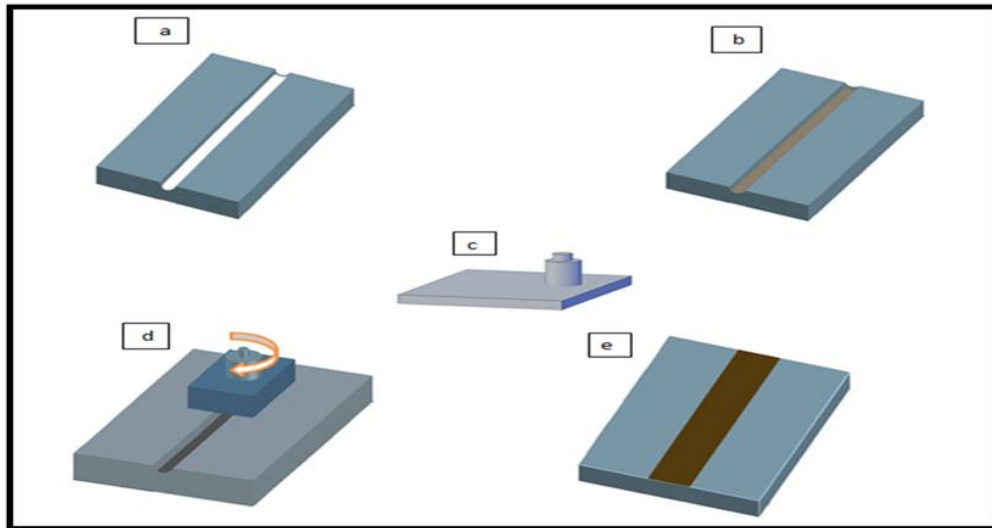


Figure (4): FSP procedure to fabricate surface composite (a) Making the Groove on the Surface of Polymer, (b) Filling the Groove with Partials, c. Shoe Tool, (d) Apply FSP on Surface by Shoe Tool, (e) Polymer Composite Surface After FSP

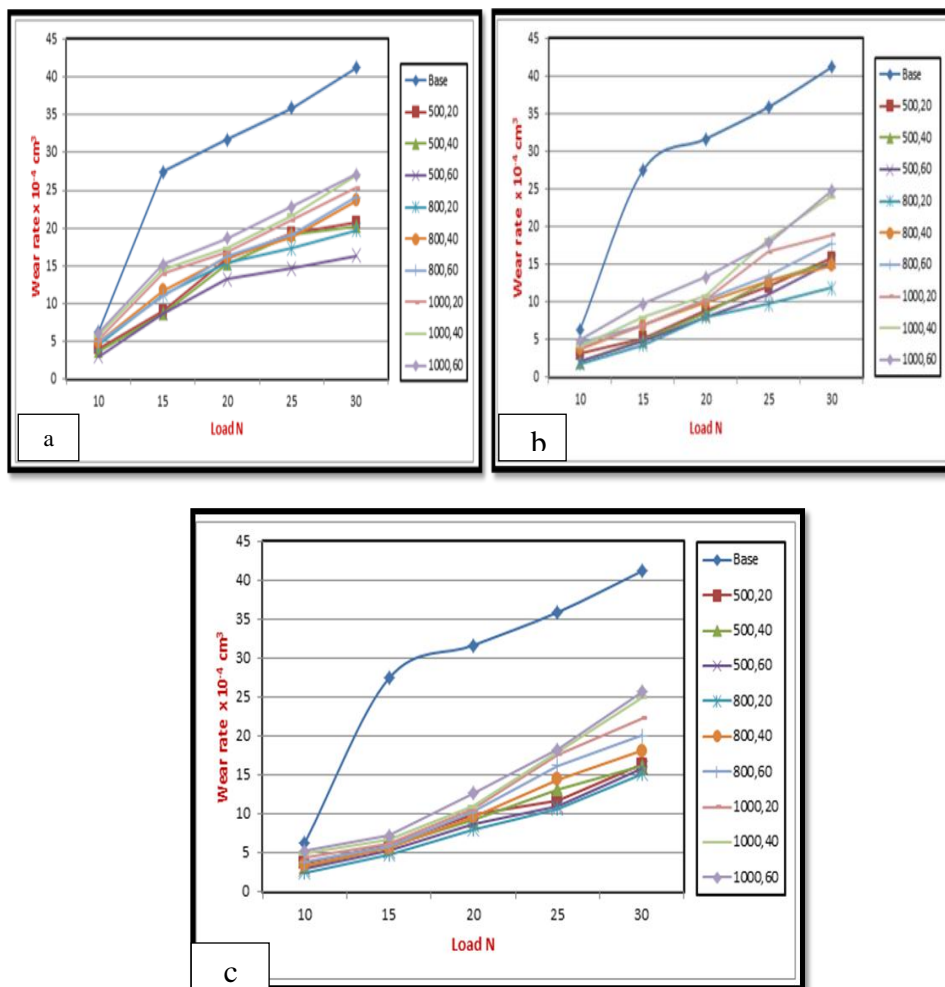


Figure (5):Wear rate at different rotational (500,800 and 1000) rpm and transvers speeds (20,40 and 60) mm/min of HDPE reinforced with (a) 5% B_4C (b) 10% B_4C (c) 15% B_4C

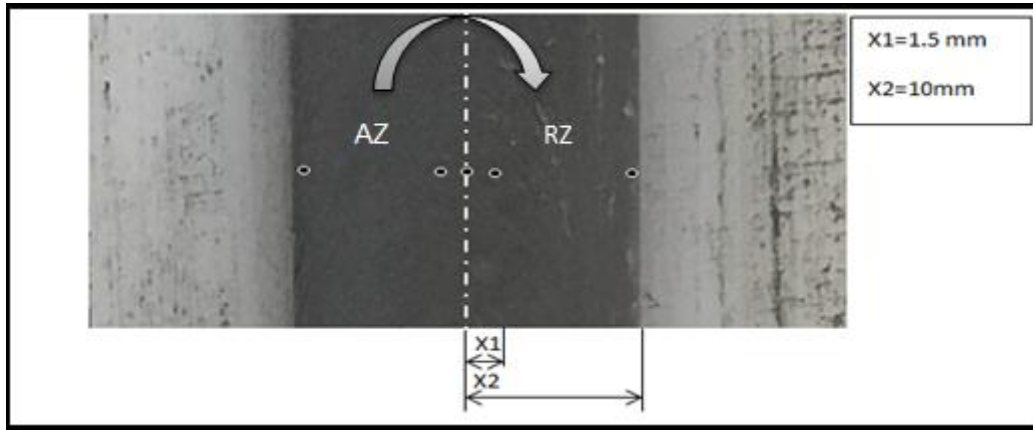


Figure (6): The Position of Point which Hardness Test In FSPed zone in HDPE/surface composite

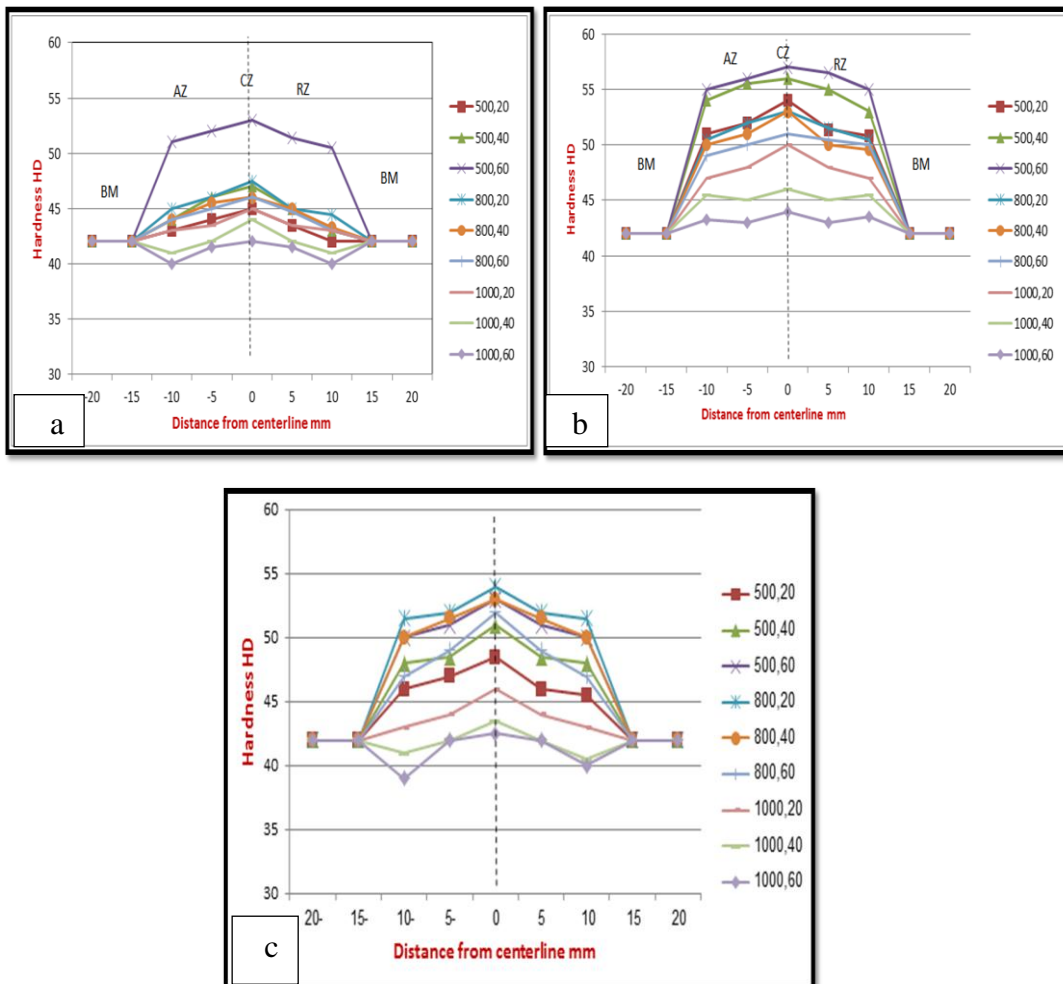


Figure (7): Hardness Profile along Stir Zone of HDPE Reinforced with (a) 5% B_4C after FSP, (b) 10% B_4C after FSP, (c) 15% B_4C after FSP were Applied with at different rotational (500,800 and 1000) rpm and transvers speeds (20,40 and 60) mm/min.



Figure (8): Optical Photomicrograph (400 X) of HDPE/ surface Composite With 10% B₄C at 800 rpm and 20mm (a) at RZ, (b) at AZ (c) at CZ.

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