Effects of Chopped Alumina Fibers on Hardness and Impact Strength of Unsaturated Polyester

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Abstract

Unsaturated polyester resins are widely used in industry by virtue of combinations of favourable characteristics including good mechanical and physical properties, ease of processing and low cost. However, these resins suffer from the disadvantage of brittleness.

In this work, polymeric composites were prepared from unsaturated polyester resin reinforced with different weight fractions of chopped alumina fibers (6%, 12%, and 18%).The following properties were studied: hardness, impact strength, and material toughness.

Experimental data showed that incorporation of alumina fiber reinforcements in the extremely brittle unsaturated polyester resin resulted in improvement material properties. Hardness, impact strength, and material toughness increased by 151%, 521% and 474% respectively at weight fraction of chopped alumina fibers (18%). The role of fiber reinforcing on the materials properties was discussed in terms of fibers nature and content.

Main Words: Polymer matrix composite, alumina fibers, mechanical properties, unsaturated polyester, fibrous composite

الخلاصة

تستخدم راتنجات البولي أستر غير المشبع بصورة واسعة في الصناعة وذلك لامتلاكها العديد من المزايا المرغوبة والتي تشمل الخواص الميكانيكية والفيزيائية الجيدة وسهولة التشكيل وقلة الكلفة. لكن إحدى مساوئ هذه الراتنجات هي صفة التقصف التي تحدد من استعمالها في العديد من الاستخدامات.

تم في هذا البحث تحضير مواد مركبة بوليمرية من راتنج البولي أستر غير المشبع مع تقويته بكسور وزنيه مختلفة من ألياف الألومينا المقطعة (6% ,12%,18%) وتم فحص خواص الصلادة, مقاومة الصدم, متانة المادة.

لقد بينت النتائج إن تقوية راتنج البولي أستر غير المشبع القصيف بألياف الألومينا يعمل على تحسين خواص المادة. إذ ارتفعت الصلادة , مقاومة الصدم, متانة المادة بنسبة(151%, 521%, 474%) على التوالي عند التقوية بألياف الألومينا وبكسر وزني(18%). تم مناقشة تأثير ألياف التقوية على خواص المادة المركبة وحسب طبيعة الألياف ومحتواها.

الكلمات الرئيسية: المواد المركبة ذات أساس بوليمري, ألياف الألومينا, الخواص الميكانيكية, راتنج البولي أستر غير المشبع, المواد المركبة الليفية.

1. Introduction

Composites are typically used in applications where a material of high strength and low weight is desired. For instance, while metals typically have high strength, they are among the heaviest materials. Plastics, while very light, tend to be comparatively weak. Ceramics in fiber form, while very strong, lack rigidity without additional support. Then, if these ceramic fibers are placed in plastics, the fibers can carry most of the force, while the plastic helps the fibers maintain the desired useful shape (Okhuysen, 2002).

Polymers are particularly attractive as matrix materials because of their relatively easy processibility, low density, good mechanical properties, and often-good dielectric properties. High temperature resins for use in composites are of particular interest to the high-speed aircraft, rocket, space, and electronic fields. It is softening temperature, its oxidative resistance, or its intrinsic thermal breakdown determines the temperature capability of a matrix resin(Feldman, 2003).

Further, new and high performance fibers reinforced polymer matrix composites are expected to satisfy many requirements for a wide range of performance-driven, and price sensitive, applications in structural applications. Fibers reinforced polymer matrix composites consists of a uniform distribution of

strengthening ceramic fibers embedded within a polymer matrix. In general, these materials exhibit good strength and stiffness, in addition to lower density. Fiber reinforced polymer matrix composites benefits from the ceramics ability to withstand high velocity impacts, and the high toughness of the polymer matrix, which helps in preventing total shattering. This contribution leads to an excellent balance between cost and mechanical properties, which are appealing for many applications (Gupta et. al., 2001).

The main contribution to the strengthening of fiber reinforced polymer matrix composites is fiber addition, which most of the mechanical properties of fiber reinforced polymer matrix composites. Several fiber parameters, which are critical in determining the mechanical properties of fiber reinforced polymer matrix composites, include the volume fraction (v_f), size, shape, and distribution of reinforcing fibers within the polymer matrix. The most influential among these parameters is (v_f) (Harper, 2000).

The aim of this work is to prepare and to study the mechanical properties (impact resistance and Vickers micro hardness) of unsaturated polyester matrix composite material reinforced by (6, 12, and 18 wt. %) chopped alumina fibers.

2.Material Used

2.1 Alumina Fibers

A need for higher modulus of elasticity, melting point, and exceptional resistance to corrosive conditions resulted in manufacturing fibers of pure or near pure Al_2O_3. These relatively pure alumina fibers contain very little if any SiO_2 and other components within the fibers composition. As the temperature continues to increase, the alumina becomes crystalline, undergoing different phase changes until it reaches the desired stable α phase. The first Al_2O_3 phase to form is η and, as the temperature decrease, changes and continues through to γ, δ, Θ, and finally the composition becomes α phase (Reinhart, 1989). The α phase of Al_2O_3 is desired for fibers used in composites with high temperature applications due to its higher temperatures resistance and modulus value (Cooke, 1991). Table 1 shows the properties of alumina and a various fiber materials (Okhuysen, 2002).

3. Matrix Material

The properties of the composite depend largely on the combination of the properties of the matrix and the fibers. Table 2 shows selected properties of the unsaturated polyester and various types of matrix materials.

4. Theoretical View

Fracture mechanics can be defined as material breaks into two parts due to the effects of external forces. Griffith was the first person who fixed the basic principles of fracture mechanics by energy balance theory. Griffith (Boresl and Schmidt, 2002)studies the process, which occurred to the crack by reverse thermodynamics process. He proposed the existence of static equilibrium condition for crack through the effects of energies given by equations 1 and 2:

$$U = (-W_L + U_o) + U_S \qquad (1)$$

Where $W_{L:}$ External performed work on system.

U_o: Saved energy in system.

U_S: Required energy to create new surface (free surface energy).

U: External impact energy.

Therefore, fracture mechanics is calculated from impact energy (U) if engineering sample dimensions and crack depth (a) are measured. When crack growth occurred,

malleability will increase for material according to the value of saved energy, therefore if malleability changes and crack depth (a) are known, absorbed energy is calculated by the following equation (Boresl and Schmidt, 2002):

$$U = \frac{1}{2} P^2 C \qquad (2)$$

Where P: applied load in Newton
C: malleability.
If sample width is B in meter then:

$$G_c = \frac{1}{B}\frac{dU}{da} \qquad (3)$$

$$G_c = \frac{1}{B}\frac{dU}{d\mathbf{C}}\frac{dC}{da} \qquad (4)$$

Where: G_C is material toughness (kJ/ m^2) when crack occurred. ($G_{C)}$ is one of the material properties, and it is regarded as absorbed energy by crack unit area, also it represents released critical strain energy, which is given by the following equation (Boresl and Schmidt, 2002):

$$G_c = \frac{P^2}{2B}\frac{dC}{da} \qquad (5)$$

From equations, two and five, absorbed energy is calculated by impact test:

$$U = G_C B \frac{C}{dC/da} \qquad (6)$$

Practically, U is calculated for different crack depths.

$$G_c = \frac{U}{BDC}\frac{dC}{d(a/D)} \qquad (7)$$

Where D: Sample thickness in meter.
So, engineering configuration function Φ is given by malleability C:

$$\Phi = \frac{C}{dC/d(a/D)} \qquad (8)$$

So equation 8 can be solved to calculate material toughness G_C:

$$U = G_C BD\Phi \qquad (9)$$

Where : $\boldsymbol{\Phi = 0.135\,(a/D)^{-0.77}}$ **(10)**

The relationship between absorbed energy (U) and engineering configuration function (Φ) is linear, and the slop of this line is material toughness $\mathbf{G_C}$ (Boresl and Schmidt, 2002).

5. Experimental Work

5.1. Alumina Fiber Preparation

The route of ceramic fibers involves the preparation of a concentrated solution with which to impregnate a conventional rayon or cotton thread chosen for its wicking rate. During heating two processes occur; the first, solution is rapidly crystallized when solvent molecules are flash- evaporated, and the second cotton is pyrolysed, leaving behind a skeleton of the relevant oxide. This pyrolysis has to be undertaken carefully to avoid disruption, and is responsible for the hollow tube morphology. The evaporation stage remove solution from the surface to cause a degree of back-wicking leaving a center core with no solution. The more rapidly the evaporation can be achieved, the smaller will be the cross section of the hollow tube.

The manufacturing process of alumina fiber is proprietary, although certain details have been made available which outline the general fabrication route. The innovation fabrication technique involves the use of an organic precursor fiber, rayon as an internal former. The organic fiber is impregnated with an aqueous solution of aluminum chloride. During drying, the metallic salts are deposited within the organic fiber, which can be burnt off by controlled oxidation.

These steps achieved the fabrication process:

1. Preparation the solution of the aluminum oxychloride in a concentration of 1- M solution.
2. Selection cotton as a substrate.
3. Impregnation of the cotton from the solution three days and extraction the cotton from the drying in furnace at 100 °C.
4. Heat treatment was achieved at two stages, heat-treated to a temperature 350 °C slowly (20°C/min.), need four hrs. to reach this temperature then raised the temperature to 600 °C (20°C/min.) and leave the cotton at this temperature to 1 hr. then slowly cooled at furnace.

5.2. Samples Preparation

The resin used in this work is Viapal H 265 unsaturated polyester resin based on tetrahydrophthalic acid and appropriate blends of ethylene glycol, propylene glycol, and di (propylene glycol) dissolved in styrene. Vianova Kunstharz, Austria supplied the resin, promoter and catalyst. Bulk resin sheets were prepared by mixing resin with 0.5% (w/w) cobalt octoate in xylene containing 6% active cobalt as promoter, and 2% (w/w) methyl ethyl ketone peroxide as a catalyst. These materials were thoroughly mixed and stirred at low speed until it become uniform. After that, the reinforcement alumina chopped fibers were mixed with the matrix resin. The composite material was poured into the mould slowly in order to avoid air trapping. The composite material was cured at room temperature until it was dry. The same steps were used to make an un reinforced unsaturated polyester.

The amounts of reinforcement fibers were calculated according to the following Equation (Lukkassen and Meidell, 2003):

$$\Psi = (W_f / W_T)\ 100\% \qquad (11)$$

Wherre Ψ: Weight fraction of fibers %.

W_f : Weight of fibers.

W_T: Total weight of sample.

The mixture was left for two hours so that it becomes a little tacky.

5.3. Mechanical Testing

5.3.1. Impact Test Samples

This test is important to calculate the energy density adsorption due to impact energy acting by kinetic energy of hammer. Hammer with 2 joules energy was used to test un reinforced polyester specimen, 10 joules energy hammer was used for fiber reinforced specimens. Standard dimensions (10* 10* 55 mm) were used to prepare Charpy impact samples according to (ISO.179) with different notch depths (0.8, 1.6, and 2.5 mm) . Unsaturated polyester samples without and with reinforcement alumina fibers at (6, 12, 18 wt. %) were tested.

5.3.2.Hardness Test Samples

All samples were subjected to Brinell hardness test using Leybold Harris multi purposes tester No. 36110 with stainless steel ball (5 mm diameter) and applied load

(10 kg) for (15 sec.). Then average indentation diameter (d) was recorded. Brinell hardness was calculated according to the following eq. (Askel and Phule, 2003):

$$HB=0.102*2*F/\pi D (D-(D^2- d^2)^{0.5} \quad (12)$$

Where F: Applied load (N).
D: Ball diameter (mm).
d: Indentation diameter(mm).

Brinell hardness test was repeated several times to evaluate Brinell hardness average value.

6. Results and Discussion

Figure 1 illustrates light optical microscopy image of chopped alumina fibers prepared in this work. Whereas Figures 2 and 3 shows diffractograms from the chopped alumina fibers and the standard diffractograms from the α-Al_2O_3 respectively.

Fig. 4 illustrate that the hardness of unsaturated polyester increases proportionality with increase of chopped alumina fibers weight fraction. This is due to diffusion of reinforcement chopped fibers between the polymer chains results in movement inhibition of these chains that increases its stability.

Result showed that Brinell hardness number of composite material reaches maximum value (73) at 18 wt. % alumina fibers. It increases 151% in comparison with unreinforced polyester. This is belongs to the good bonding between the alumina fibers and matrix material, then the hardness is, roughly speaking, an average of those of the matrix and fibers, weighed by their weight fractions. The interface is crucial in controlling composite properties because load is transferred from the matrix to the fiber through the interface (Flinn and Trojan, 1981). These results are in a good agreement with those obtained by Shaker et. al. (Shaker et. al. , 2010). They found that the addition of zirconia fibers to unsaturated polyester resin in the 5 wt.% , hardness increasing by 41%.

In this work, composites with chopped alumina fibers, the critical factors are the length –to-diameter ratio of the fiber, the shear strength of the bond between the fiber and the matrix, and the amount of fibers. All these variables affect the strength of the composite. However, a good deal of composites are made with chopped –up pieces of alumina because of the ease with which they are formed into complex shapes even when the length to diameter ratio is unfavorable (Flinn and Trojan, 1981).

Fig. 5 illustrate the effect of added alumina fibers in different weight fractions (6, 12, 18 wt. %) to unsaturated polyester on fracture energy (joule). It has concluded that fracture energy was increased with increasing fiber weight fractions up to (18 wt. %). Reinforcement fibers act as strong obstacle against cracks propagation and this will tend to change crack shape and direction. Then this crack will transform into secondary cracks. Change in crack shape and direction leads to increase crack surface area and consumed fracture energy. All these factors tend to increase composite strength especially in case of good bonding between reinforced fibers and matrix material (Hanan et. al., 2005).

Results indicated that impact resistance was increased by a ratio of (521%) with increasing alumina fibers weight fraction at (18 wt. %). These results are in a good agreement with those obtained by Elie (Elie , 2007). He found that the addition of alumina particles to unsaturated polyester resin in the 20 wt.% , impact resistance increasing by 45%.

Further, fig. 5 showed impact energy (U) variation with engineering configuration function (BDΦ) by variable crack depth (a) under different chopped

alumina fibers at a selected weight fraction of (6, 12, 18) %. It's clear that fracture impact energy was decreased with increasing of crack depths due to decreasing cross section area of sample under shock test.

During preparation of current composite material, unsaturated polyester as a continuous phase, envelops the alumina fibers and forms a coherent bond, usually chemical in nature, at the fiber/ matrix interface. During deformation, the matrix transfers stress to the fibers, which are usually have a higher elastic modulus. The response of the composite then depends on the relative properties of the fiber and the matrix, the weight fraction of each phase, the fiber length and orientation. Failure of individual fibers is local and takes place without destroying the integrity of the whole composite. If debonding of the matrix/fiber interface takes place because of a propagating crack, a significant amount of strain energy is required ((Flinn and Trojan, 1981).

Table 3 showed material toughness of prepared samples. The toughness G_C of composite (like that of any other material) is a measure of the energy absorbed per unit crack area. Material toughness (G_C) was calculated for each sample from the slop of linear relationship between fracture energy (U) and engineering configuration function BDΦ (as in fig.4). Results showed that maximum toughness was found in case of reinforced unsaturated polyester by 18 wt. % alumina fibers (86.7 kJ/m^2).

7. Conclusions

This research covers reinforcement of unsaturated polyester , and focalizes on studying Brinell hardness number, impact resistance, and material toughness properties by preparing the unsaturated polyester reinforced by random chopped alumina fibers at a selected weight fraction of (6, 12, 18) % . The following results can be concluded:

1. Addition of alumina fibers to unsaturated polyester increase Brinell hardness number proportionality especially in case of reinforcement by weight fraction at (18 wt. %), it increases 151% in comparison with unreinforced polyester.
2. The toughness G_C of prepared composite material reaches maximum value with increasing alumina fibers weight fraction at (18 wt. %), it increased by a ratio of 474 % in comparison with unreinforced polyester.

8. References

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Table 1: Properties of Fiber Materials (Okhuysen, 2002)

Fibers	Density (g/cm^3)	Tensile Strength(MPa)	Modulus of Elasticity (GPa)	Thermal Expansion (10^{-6}/°K)
E- glass	2.55	3440	72.3	
S-glass	2.50	4480	86.8	
Alumina	3.15	2070	172.1	7.0
Graphite (High Strength)	1.50	2760	275.4	
Graphite (High Modulus)	1.50	1860	530.2	

Table 2: Properties of Polymer Matrix Materials (Okhuysen, 2002)

Materials	Density (g/cm^3)	Tensile Strength (MPa)	Modulus of Elasticity (GPa)	Thermal Expansion (10^{-6}/K)
Polyester	1.1-1.4	34.5-103.5	2-4.4	55-100
Epoxy	1.2-1.3	55-130	2.75-4.1	45-65
Polyimide	1.46	120	3.5-4.5	90
Phenolic	1.30	50-55	-	45-110

Table 3: Material toughness values for Unsaturated Polyester (U.P) samples with and without reinforcement alumina fibers at (6%, 12, 18 wt. %).

Material	Toughness (kJ /m^2)
Unsaturated Polyester(U.P)	15.1
U.P+6%Al2O3	59.4
U.P+12%Al2O3	64.3
U.P+18%Al2O3	86.7

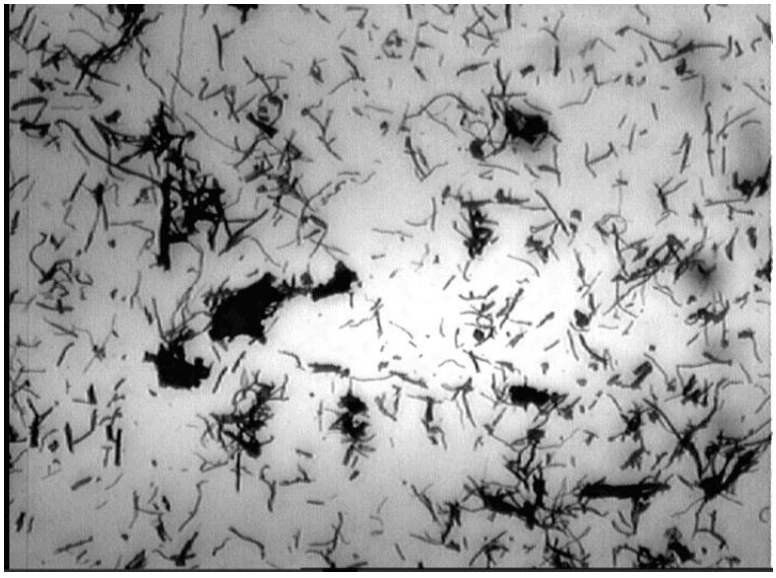


Figure 1: Light optical microscopy images of chopped alumina fibers which prepared in this work(Magnification 400X).

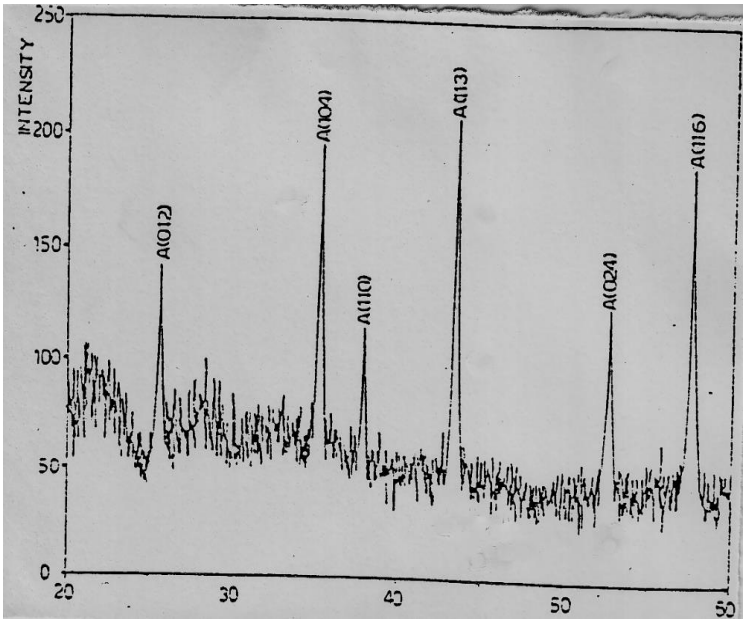
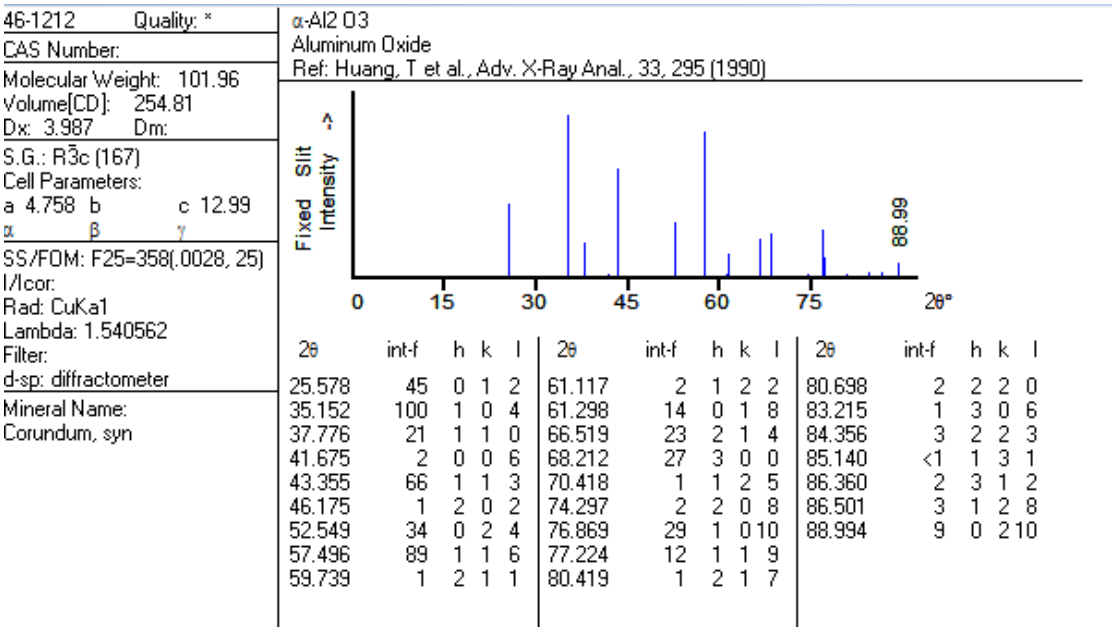


Fig. 2: Diffractograms from the surface of chopped alumina fibers (Al_2O_3)



46-1212 Quality: *
CAS Number:
Molecular Weight: 101.96
Volume[CD]: 254.81
Dx: 3.987 Dm:
S.G.: R$\bar{3}$c (167)
Cell Parameters:
a 4.758 b c 12.99
α β γ
SS/FOM: F25=358(.0028, 25)
I/Icor:
Rad: CuKa1
Lambda: 1.540562
Filter:
d-sp: diffractometer
Mineral Name:
Corundum, syn

α-Al2 O3
Aluminum Oxide
Ref: Huang, T et al., Adv. X-Ray Anal., 33, 295 (1990)

Fixed Slit Intensity

88.99

0 15 30 45 60 75 2θ°

2θ	int-f	h	k	l	2θ	int-f	h	k	l	2θ	int-f	h	k	l
25.578	45	0	1	2	61.117	2	1	2	2	80.698	2	2	2	0
35.152	100	1	0	4	61.298	14	0	1	8	83.215	1	3	0	6
37.776	21	1	1	0	66.519	23	2	1	4	84.356	3	2	2	3
41.675	2	0	0	6	68.212	27	3	0	0	85.140	<1	1	3	1
43.355	66	1	1	3	70.418	1	1	2	5	86.360	2	3	1	2
46.175	1	2	0	2	74.297	2	2	0	8	86.501	3	1	2	8
52.549	34	0	2	4	76.869	29	1	0	10	88.994	9	0	2	10
57.496	89	1	1	6	77.224	12	1	1	9					
59.739	1	2	1	1	80.419	1	2	1	7					

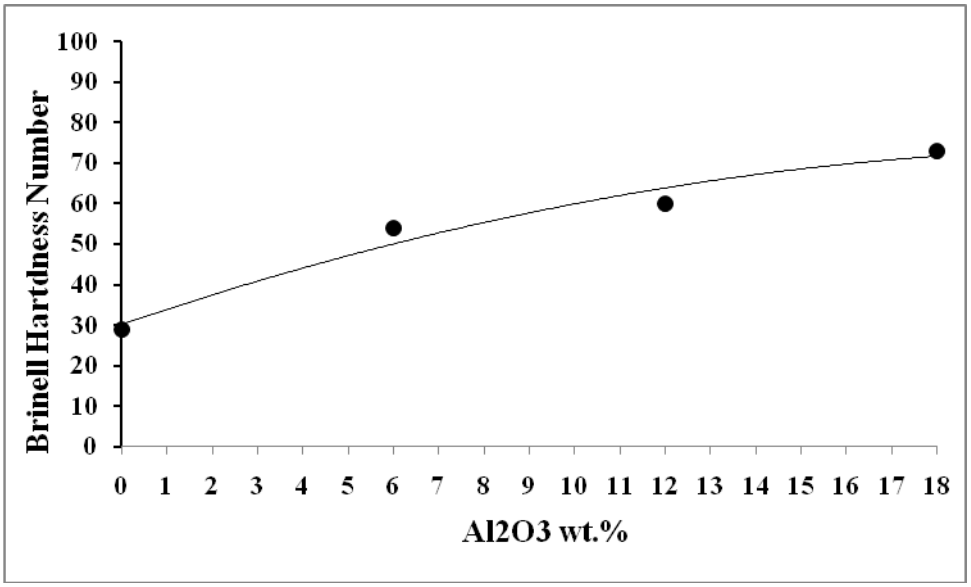


Fig. 3: Illustrate the relation between concentration of α-alumina fibers and Brinell hardness number.

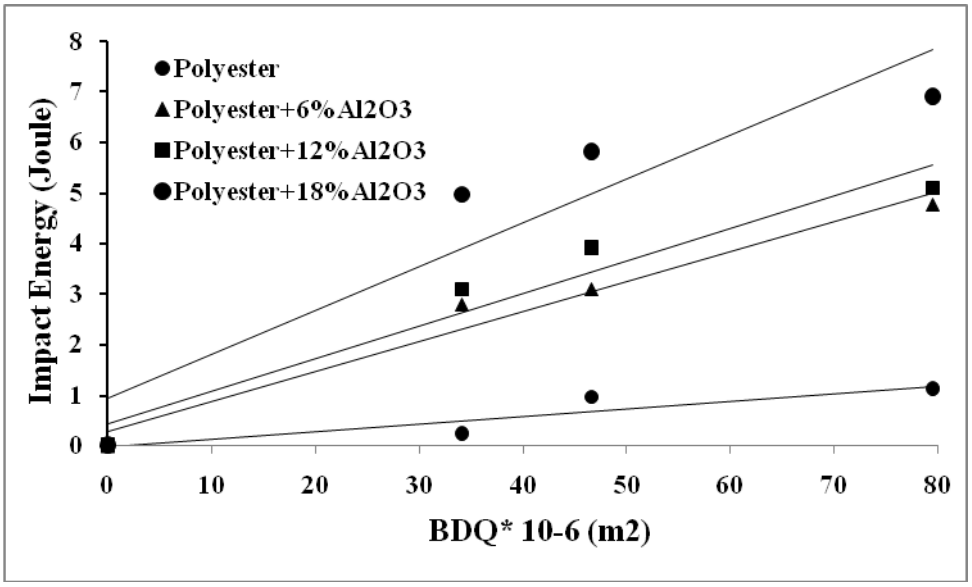


Fig. 4: Illustrate the relation between impact energy and BDΦ