THE APPLICATION OF INSTRUMENTED FALLING WEIGHT IMPACT TEST TO DETERMINE THE DUCTILE-BRITTLE TRANSITION IN ACRYLATE RUBBER-MODIFIED PVC

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Abstract

The current trend in window frame application shows the importance and popularity of high impact-acrylate modified PVC. The objective of this research is to extend the existing knowledge of toughness enhancement in rubber-toughened polymer materials to acrylate rubber-modified PVC, similar to compounds used in window frame applications. This paper reports the use an instrumented falling weight impact test (IFWI) method to determine ductile brittle transitions in acrylate rubber-modified PVC-U. The results show that force and energy data from the IFWI test were sensitive to the filter level and an optimum filter level for apparatus/specimen combination is thus necessary. The IFWI tests have highlighted the difficulties which must be overcome to obtain accurate and reliable data in the determination of impact strength values at high impact velocities, for brittle specimens. The results also show that over the impact velocity range studied (i.e. 1 to 6 m/s) all the acrylate rubber-toughened PVC blends except blend M2 showed a transition from ductile to brittle at a temperature of around 20°C. The impact modifiers were found to differ in their efficiency to shift the ductile to brittle transition.

Introduction and Objectives

It has been observed that normally-ductile materials often fail prematurely through brittle fracture. The transition from ductile to brittle failure occurs when the yield strength is equal to brittle strength (1). In high-loading conditions, parameters such as temperature and strain rate become more important due to the viscoelastic nature of polymers. Unnotched unplasticised PVC (PVC-U) specimens show considerable ductility in slow rate tension tests but when notched and tested under high-loading rate they fail in a relatively brittle manner with low impact strength. With the presence of rubber-based polymer in PVC, the yield strength decreases thus improving the impact strength of the rubber-toughened polymers resulting in the shift of the ductile-brittle point to a more severe condition (1).

The current trend in window frame application shows the importance and popularity of high impact-acrylate modified PVC (2). The objective of this research is to extend the existing knowledge of toughness enhancement in rubber-toughened polymer materials to acrylate rubber-modified PVC, similar to compounds used in window frame applications.

This paper reports the use an instrumented falling weight impact test (IFWI) method to determine ductile brittle transitions in acrylate rubber-modified PVC-U and to study the effect of strain rate. The effect of filter level on peak energy, failure energy and peak force were also investigated using the IFWI

Materials and Methods

The PVC used in this study is a suspension resin with solution viscosity K-value 68, Evipol SH 6830, supplied by EVC. The acrylate-grafted PVC copolymer is Vinidur SZ (solution viscosity K-value 66) supplied by BASF with 6 % rubber content. The additives used are shown in Table 1.

Four different types of core-shell acrylic impact modifiers were used in this study. Three modifiers, referred to here as A, B, and C are of the core and shell type particles which were thought to differ in their rubber particle size and core to shell ratio. The outer shell is made up of PMMA which is compatible with PVC and the core consists of polybutyl acrylate (PBA) rubber. The other modifier referred to as D is acrylate rubber grafted to the PVC, i.e. as a rubber phase in a copolymer.

Table 1: Types and Suppliers of Additives

Additives	Types	Suppliers
Impact Modifier	Core-shell type acrylic modifier	Rohm and Haas
Internal Lubricant/ Stabiliser	Calcium Stearate	
External Lubricant	Paraffin Wax (Irgawax 367)	Ciba-Geigy
Light Stabiliser	Tinuvin 320	Ciba-Geigy
Heat Stabiliser	Tin (Irgastab T196)	Ciba-Geigy
Processing Aids	Acrylic (PA K120N)	Rohm and Haas
Processing Aids	Acrylic (PA K175N)	Rohm and Haas

The PVC blend formulations are shown in Table 2. These are based upon a tin stabilised formulation, but without the pigment and fillers usually present in commercial window profile materials (in order to enhance optical transparency in microscopic studies).

Table 2 : Blend Formulations

	Blend				
Ingredient	M0	M1	M2	M3	M4
PVC	100	100	100	100	-
Impact Modifier A	-	6.4	-	-	-
Impact Modifier B	-	-	6.4	-	-
Impact Modifier C	-	-	-	6.4	-
PVC-Ac Copolymer	-	-	-	-	100
External Lubricant	1.5	1.5	1.5	1.5	1.5
Internal Lubricant	1.0	1.0	1.0	1.0	1.0
Light Stabiliser	1.5	1.5	1.5	1.5	1.5
Heat Stabiliser	2.0	2.0	2.0	2.0	2.0
Processing Aids	2.0	2.0	2.0	2.0	2.0

Numbers indicates parts per hundred resin (phr) in blend

In the PVC-acrylate rubber blends, 6.4 phr impact modifier was chosen to be equal to the amount of impact modifier present in the PVC-polyacrylate copolymer. The concentrations of other additives were based upon typical commercial PVC window formulations.

A high speed laboratory mixer (Fielder 8L) was used to blend PVC and the additives. PVC and the solid additives were added when the set temperature reached 80 °C. The mixer was switched on and the mix was agitated at 2000 rpm. The temperature was then reset to 125 °C. When the actual temperature of mixer reached 75 °C, the rotor speed was reduced to 1000 rpm and the stabilizer in liquid form was then added. The rotor speed was then increased to 2000 rpm. The mix is then agitated until the real mixer temperature reached 120 °C. At that temperature the speed of the rotor was reduced to 1500 rpm. The blend is then dumped into cooling chamber (Fielder 12L) and was left for 5 minutes before being collected.

To produce moulded flat sheet, the dry blends of PVC and additives are firstly sheeted on a two roll-mill. Flat sheets with dimensions of 120 mm by 120 mm and 3 mm thickness were produced by compression moulding the milled sheets at the set temperature of 185 °C. Based on calculation, enough quantity of the milled material was placed inside the mold and pressed at low pressure (6.06 MPa) for 3 minutes and high pressure (24.22 MPa) for 4.5 minutes at 185°C.

Samples for impact testing were cut from the compressed sheet according to the size specified in BS 2782: Part 3: Method 359 (1984). Dummy bars were routed to the standard size on a tungsten carbide cutter using a template. Sample dimensions were measured using vernier calipers. Notching was carried out using the broaching tool and fixture. The notches are 'dead-sharp' unless otherwise stated. The notch depth was measured using a travelling microscope.

The impact strength assessment was done using a Rosand Type 5 Instrumented Falling Weight Impact (IFWI) Tester. The Rosand type 5 uses a Kistler piezo-electric transducer which can produce (as standard) full scales from 10N to 50 000N. The impactor used has the same radius as that of the pendulum used in a conventional Charpy test. To retain consistency with the conventional Charpy test, a jig which allowed a span of 40mm was used.

The test cycle is controlled by a microprocessor system. From the moment of crack initiation the sensor measures and stores the force readings on a transient recorder and carries out calculations on these readings. Force-time data are converted into force-distance data by assuming constant velocity and from these data, failure energies, forces and deflection are calculated. The system allows certain parameters to be set before testing. Parameters which were used in all tests are listed in Table 3.

Table 3: Parameters Set on the Instrumented Falling Weight Impact Tester.

Mass (kg)	25
Delay	10
Sweep time (ms)	5 and 10
Force range (N)	0 - 200
Temperature (°C)	-40 to 80

The sweep time sets the length of time that recordings will be taken (and thus the frequency) after the data capture system has been triggered. Lowering the sweep time increases the resolution as all the data points are recorded in shorter time span. The lowest available setting is 2 ms but it was found that in certain cases this did not permit the full trace to recorded. A setting of 5 and 10 ms was found to be satisfactory and was therefore adopted as standard. Delay values range from 0-10 and indicate the number of points recorded after the trigger. 0 therefore means that all points recorded occurred before the trigger pulse was received, 10 indicates that all values collected occurred after the trigger pulse.

To investigate the effect of strain rate on impact strength, the impact velocity was varied between 1 to 6 m/s by dropping the mass at the appropriate height with the temperature kept constant at 20 °C. The relationship between the height of the impactor and the impact velocity is shown in Table 4.

Table 4: Relationship Between Impactor Height and Impact Velocity

Impactor Height (mm)	Impact Velocity (m/s)
51	1
204	2
459	3
816	4
1276	5
1837	6

Results and Discussion

The study of the effect of filter frequency on force-deflection traces in Instrumented Falling Weight Impact (IFWI) testing is important to determine the most suitable filter frequency for all future IFWI testing work. During impact there are vibrations which interfere with the true force-deflection curve and therefore filtering of the noise is necessary to remove part of the curve due to the interference. The Rosand Impact Tester has a facility whereby the signal can be electronically filtered. The frequency of the filter control can be set manually at levels between 1 and 20 kHz. For example, a setting of 1 kHz indicates that the signal is re-processed to remove all vibrations which have frequency greater than 1 kHz and hence is the 'harshest' filter. As it is possible to apply a different filter to the same original signal, the influence of filter frequency on peak force, peak energy and failure energy can be studied.

The study of the effect of filter frequency on force-deflection traces was carried out at an impact velocity of 3 m/s which is very similar to the impact velocity of Charpy Impact Testing (2.98 m/s). It was also decided that in the study of the variation of impact strength with temperature, a constant impact velocity of 3 m/s would be used. The shape of the curves changes as the filter frequency changes. Figures 1(A-D) show the effect on the shape of the curve of applying 1, 3, 5 and 7 kHz filters respectively. A low filter value such as 1 kHz gives a smooth trace which is easier to analyse. However, it may remove part of the signal which is due to the fracture of the sample. This would in turn lead to fracture values which would be unduly pessimistic. However, reducing the severity of the filter from 1 to 3 kHz reveals the presence of another peak before the maximum peak. This first peak increases with increasing filter frequency used.

The variation of data output (such as peak force, peak energy and failure energy) with filter frequency from the IFWI test of a brittle fractured sample tested, at 3 m/s was obtained. Figure 2 (A and B) present the variation of the peak force, and peak and failure energy respectively, with filter frequency. The results clearly indicate that the peak force increases with increasing filter frequency. However, it was observed that peak energy and failure energy decreased initially as the filter frequency increased from 1 kHz to 2 kHz. However, from 3 kHz to 10 kHz the values for peak energy and failure energy remain steady at around 25 mJ and 38 mJ respectively. Both the peak and failure energy also seem to reach the plateau at around a filter frequency of 3 kHz. This initial result is in agreement with the earlier finding by Calvert (3) that force and energy data were sensitive to filter level and appropriate filter level for apparatus/specimen combination is thus necessary.

From Figures 1-A and 2-B, it can be observed that for the filter frequency of 1 kHz, the peak energy (i.e. the crack initiation energy) appears to be less than the propagation energy, which is not expected for brittle failure samples. However as the filter frequency increases from 1 kHz to 3 kHz (as shown in Figure 1-B and Figure 2-B), the peak energy (i.e. the crack initiation energy) is greater than the propagation energy which is more realistic for brittle failure samples.

As mentioned earlier, the study of the effect of filter frequency on force-deflection traces in Instrumented Falling Weight Impact (IFWI) testing is to determine the most suitable filter frequency for all future IFWI testing work. The filter frequency of 3 kHz was selected for future work for the following reasons:

(a) A low filter frequency of 1 kHz is not suitable because it may remove part of the signal which is due to the fracture and will lead to fracture values which would be unduly pessimistic. The force-deflection traces obtained from tests filtered at 1 kHz also showed the propagation energy to be greater than peak energy (crack initiation energy), which is unrealistic for brittle failure samples.

(b) At filter frequencies above 1 kHz, the force-deflection traces show the presence of another peak which increases with increasing filter frequency. A high filter frequency therefore is not suitable because as the filter frequency increases, the deviation from an ideal force-deflection trace (i.e. a smooth trace) increases due to the presence of dynamic effects.

(c) A filter frequency of 3 kHz is found to be a good compromise between minimising the interference of the noise and having unrealistic and unduly pessimistic fracture values. It is also observed that both the peak and failure energy reached the plateau at filter frequency of around 3 kHz.

Having determined that the most suitable filter frequency was 3 kHz which was then used in all future studies, another study was carried out to investigate the effect of impact velocity on force-deflection traces. The shape of the curves changes with changes in impact velocity (see Figure 3). At an impact velocity of 1 m/s, a relatively smooth force-deflection trace is observed, showing that the dynamic effects are not so significant. As the impact velocity increases to 3 m/s, two peaks were observed with the first one much lower than the maximum peak. The possible causes for the first peak are the inertial loads on the sample and the oscillations in the impact system (4-8). It also appears that the proportion of propagation energy increases slightly as the impact velocity increases from 1 to 3 m/s. This is unexpected because the propagation energy should decrease with increasing impact velocity if the materials become more brittle. This may therefore influence the overall failure energy, but not to a large extent.

As impact velocity increases, it becomes increasingly difficult to interpret the resulting load-deflection traces from the IFWI tests for the brittle samples. It is also observed that the height of the first peak increases with increasing impact velocity. At impact velocities of 4 to 6 m/s (for blends M0, M1 and M2), the first peak increased to a level that is either higher or only slightly lower than the second peak, and this will have a large influence on correct determination of the total failure energy. Total failure energy is defined as the area under the curve up to the maximum peak, in a force-deflection trace (see Figure 3-B). The other difficulty with high impact velocity testing is to locate exactly the point of crack initiation. It has been shown that at high impact velocities, the onset of fracture is not automatically associated with a specific feature on the force-deflection trace (9). In particular, it does not necessarily coincide with a force peak. Therefore, it is concluded that the failure energy data obtained from IFWI tests for blends M0, M1 and M2 at impact velocities of 4 to 6 m/s are not reliable. Many previous papers (4-8) have also cited the difficulties in interpreting and analysing the resulting load-deflection traces from the IFWI testing at high impact velocities.

Table 5 shows the number of samples which failed in brittle mode as the impact velocity varied. As the impact velocity increases, generally the impact-modified samples show a decrease in impact strength. Eventually the samples undergo a ductile-brittle transition with a sharp drop in impact strength when the strain rates are high enough. It is also observed that each modifier differs in its effectiveness to shift the ductile-brittle transition to a higher strain rate. The result shows that the acrylate-modification of PVC results in the shift of the ductile-brittle transition to higher test rates, compared with that for unmodified PVC, at a given temperature.

Table 5: Variation of Brittle Fractured Samples with Impact Velocity

	Impact Velocity (m/s)					
Blends	1	2	3	4	5	6
M0	6(6) **	6(6)	6(6)	6(6)	5(5)	5(5)
M1	0(5)	7(7)	6(6)	7(7)	7(7)	6(6)
M2	8(8)	6(6)	6(6)	9(9)	5(5)	6(6)
M3	Not Done	0(6)	0(6)	0(6)	2(5)	5(5)
M4	Not Done	0(5)	0(6)	4(5)	6(6)	5(5)

** The total number of samples tested is given in bracket

From Table 5, it can also be observed that not all blends readily undergo a ductile-brittle transition in the experimental range; only blends M1, M3 and M4 undergo the transition. All samples in blends M2 and M0 failed in brittle mode and do not undergo any ductile-brittle transition over the range of strain rates studied.

As the strain rate increases, blend M1 is the first sample to undergo the ductile-brittle transition. At 1 m/s, all the samples from blend M1 fail in ductile mode exemplified by the hinge break fracture and the stress whitening effect. All the samples fail in brittle mode at 2 m/s but still show relatively high impact strength values with some of the samples indicating the existence of stress whitening at the fracture surface.

At impact velocities of 2 and 3 m/s, samples M3 and M4 fail in ductile mode completely. As the impact velocity increases to 4 m/s, samples M3 still fail in ductile mode completely whereas samples M4 fail in mix-mode with one out of five samples were observed to fail in ductile mode. At 5 m/s, samples from blend M4 failed in ductile mode completely but two out of five samples from blend M3 failed in brittle mode. Samples of blend M3 and M4 fail in brittle mode completely at 6 m/s. It can be inferred that the impact modifier C in blend M3 is the most effective in shifting the ductile-brittle transition to a higher strain rate.

The general trend which can be observed is that impact modifier C (in blend M3) is the most effective in shifting the ductile-brittle transition to a higher strain rate. This is closely followed by impact modifier D (in blend 4). The next most effective impact modifier is impact modifier A (in blend M1) and the least effective is impact modifier B (in blend M2). These results are quoted on the basis of the addition levels used (see Table 2).

As mentioned earlier, the present study has highlighted some problems in using IFWI test to obtain reliable failure energy data at high impact velocities. During any impact event, the specimen under test and the striker react to the impact oscillations in a number of ways (4). Firstly, the specimen and striker may momentarily lose contact as the specimen begins to deform so that load signal recorded by the instrumentation will initially fall and then rise as the specimen is re-loaded. Secondly, once the specimen and striker are in contact they still may oscillate but this time under a forced vibration regime.

The inertial load which is present in all impact situations depends on the requirement to accelerate the stationary specimen to the velocity of the striker (4). From the study made by Zanichelli *et al.* (5), it has been proposed that the first peak generally observed in the recorded force-time curve reflects a purely inertial load effect and involves only the part of the test specimen around the area of contact with the striker. It has been shown that the magnitude of this inertial load is proportional to the impact velocity and becomes more pronounced when operating at greater impact velocities and as such, can pose a serious problem during data analysis.

At impact speeds of less than 1 m/s the dynamic effects may be negligible and at speeds around 1 m/s the dynamic effects may become significant but still controllable (6). At speeds of several meters per second and higher, the dynamic effects become dominant. When this is the case, a rigorous evaluation of failure energy merely from the force-deflection trace becomes extremely difficult. Two alternative approaches have been proposed by various researchers to overcome the problem (8):

(b) Reduce the dynamic effects to an insignifcant level, e.g. by mechanical and electrical filtering.
(a) Take effects into account using a thorough analysis of the measured data, by proposing mechanical models (e.g. spring-mass model) to simulate the process.

In the present study, electrical filtering with a filter frequency of 3 kHz was used in the IFWI tests at all impact velocities. The force-deflection traces given in Figure 3 show that at an impact velocity of 1 m/s, a relatively smooth force-deflection trace is observed. As the impact velocity increases to 3 m/s two peaks were observed, with the first one much lower than the maximum peak. The first peak increases with increasing velocity and at impact velocities 4 to 6 m/s, the first peak is higher than the second peak. The increase of the first peak with increasing impact velocity has also been mentioned by Zanichelli *et al.* (5) and he proposed that the first peak in the force-deflection traces is due to the inertial load.

Electrical filtering was also used by Satoh and Takahashi (10) who showed that a 3 kHz filter frequency provides better results for impact tests of engineering plastics than lower, or higher frequencies. A drawback of this technique however is that the inertial load effect and the effect of specimen vibration cannot be avoided when they are intense, which will influence the fracture energy measurements (8). The results from the present study have also shown that a filter frequency of 3 kHz is not effective in reducing the inertial load effect and the effect of specimen vibration at high impact velocity.

Another method that was used to reduce the dynamic effects during high velocity impact testing is the mechanical filtering. Aggag and Takahashi (7) applied a thin rubber sheet to the impact surface of a specimen to damp the dynamic effect. It was shown from the results for polycarbonate Charpy specimens that mechanical filtering (for a thickness of 0.5 and 1.0 mm) gives good reliability at impact velocities of both 2.8 and 4.0 m/s, not only in reducing the inertial oscillation effect, but also in providing correct values of impact energy. Mills and Zhang (11) have also investigated the causes of the force oscillations observed in instrumented impact tests and suggested that the initial cause of these oscillations is the impact between the steel striker and the specimen. They found the magnitude of the initial peak to be proportional to the impact velocity, similar to the results in the present study. Their studies have also showed that one effective method of removing oscillations from instrumented impact tests is to reduce the contact stiffness impact between the steel striker and the specimen using a low modulus material of low coefficient of restitution, such as polyurethene rubber. This was found to produce more meaningful force-deflection or stress-strain curves without changing the values of the failure stress. A similar approach could be considered for future work on impact-modified PVC-U.

Conclusions

The main conclusions which can be drawn from this study are as follows:

(1) Force and energy data from the IFWI test were sensitive to the filter level and an optimum filter level for apparatus/specimen combination is thus necessary. It was found that peak force increases with increasing filter frequency. The failure energy increases with increasing filter frequency until a steady value is reached. A filter frequency of 3 kHz is found to be the optimum value for apparatus/specimen combination at an impact velocity of 3 m/s.

(2) The IFWI tests have highlighted the difficulties which must be overcome to obtain accurate and reliable data in the determination of impact strength values at high impact velocities, for brittle specimens. It was found that a filter frequency of 3 kHz is not effective in reducing the inertial load effect and the effect of specimen vibration at impact velocities greater than 3 m/s. Therefore, a filter frequency greater than 3 kHz is necessary to overcome the problem and the optimum frequency will be different for different velocities.

(3) Over the impact velocity range studied (i.e. 1 to 6 m/s) all the acrylate rubber-toughened PVC blends except blend M2 showed a transition from ductile to brittle at a temperature of around 20°C.

(4) The IFWI tests show that the chances of brittle failures in acrylate rubber toughened PVC can be reduced by reducing the strain rate. The IFWI tests also show that the impact modifiers differ in their efficiency to shift the ductile to brittle transition.

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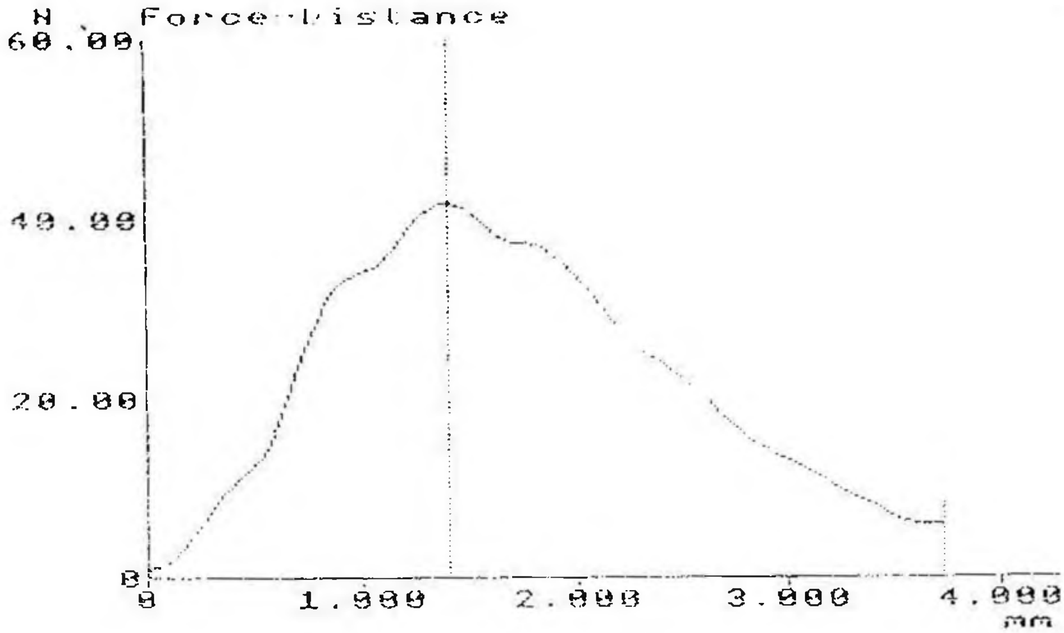
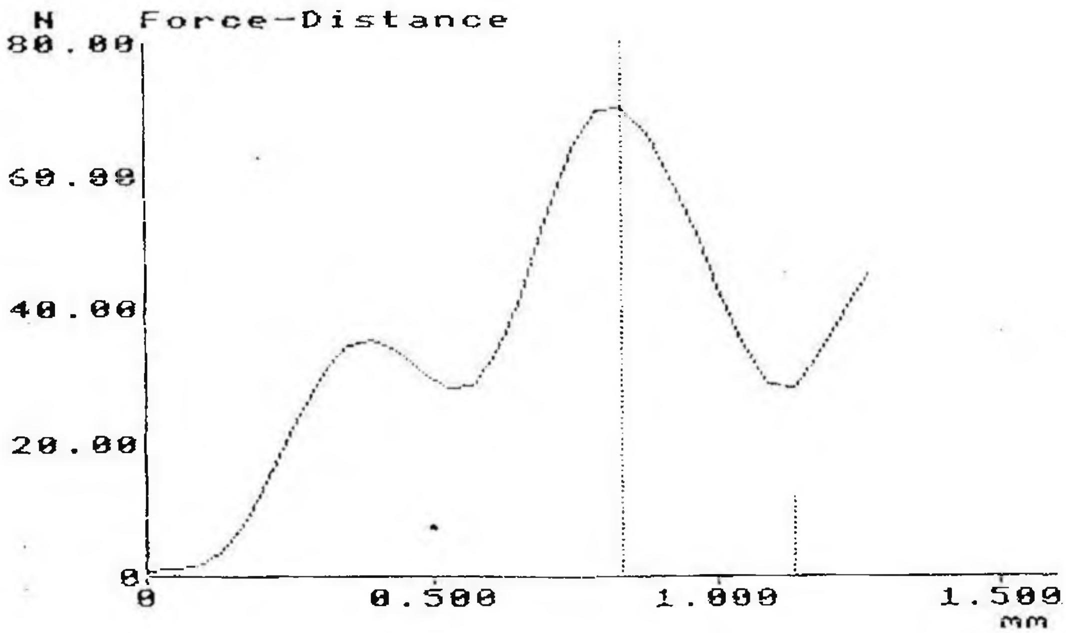
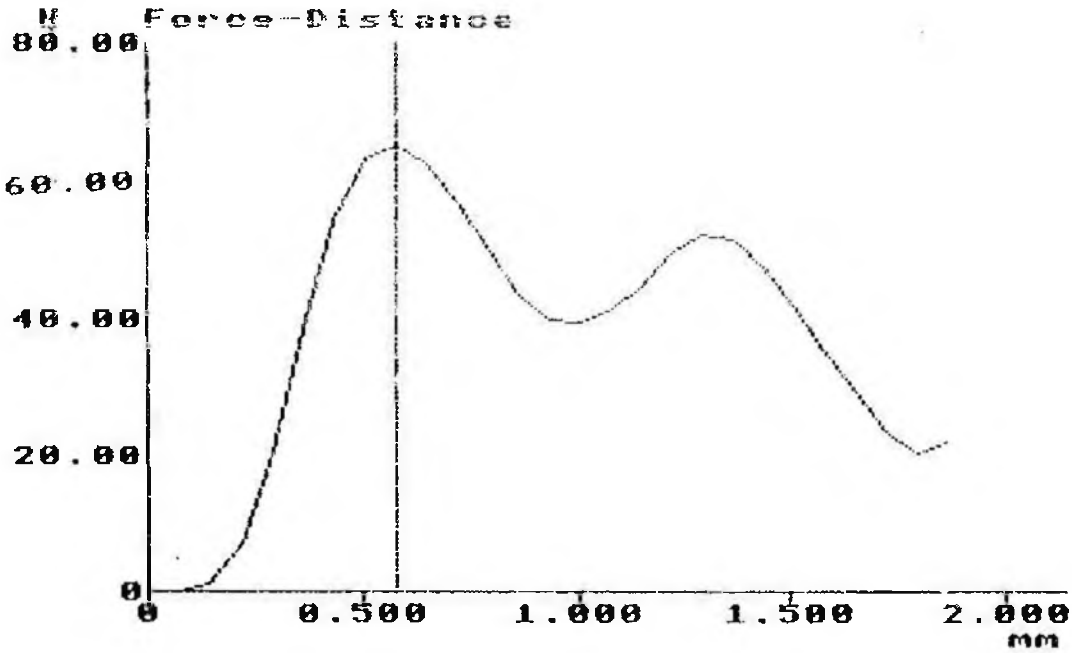
Figure 1: Effect of Filter Frequency on the Force Deflection Curve



(A) 1 Khz



(B) 3 Khz


N
Force-Distance
80.00
60.00
40.00
20.00
0
0
0.500
1.000
1.500
2.000
mm

(C) 4-6 m/s

Figure 2-A: Effect of Filter Frequency on Peak Force

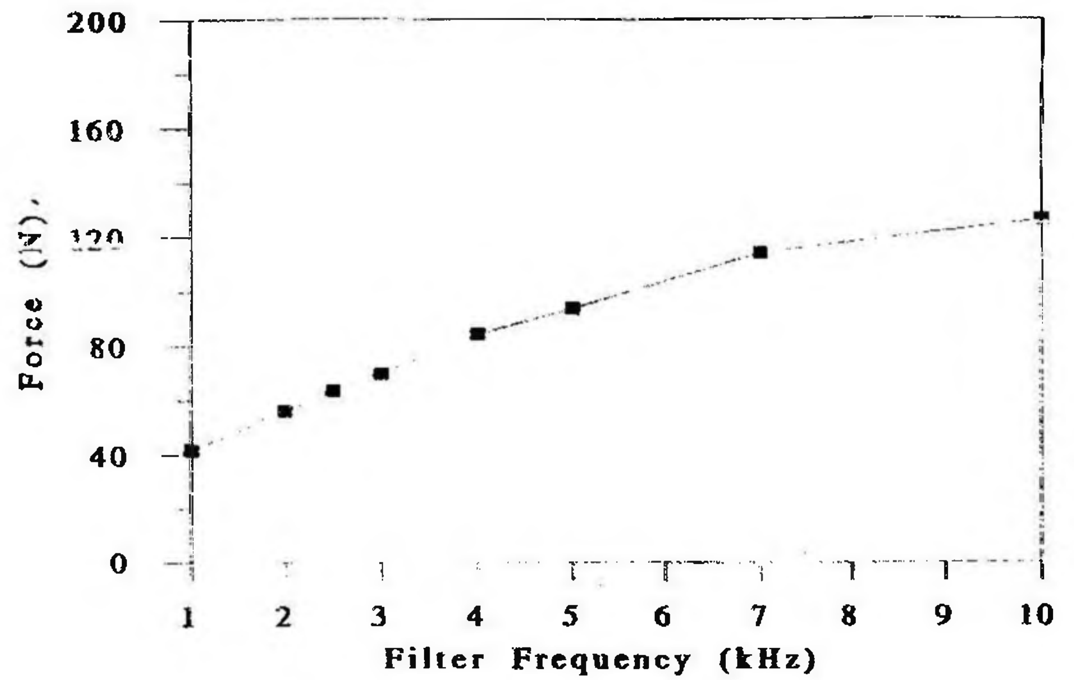


Figure 2-B: Effect of Filter Frequency on Peak and Failure Energy

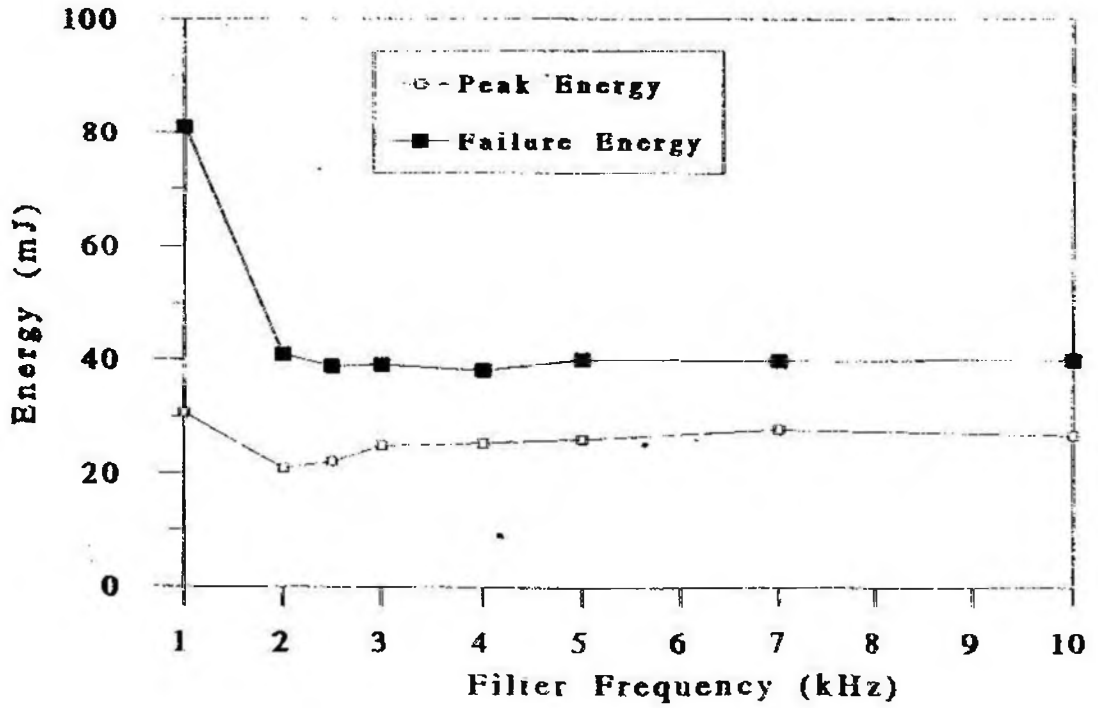
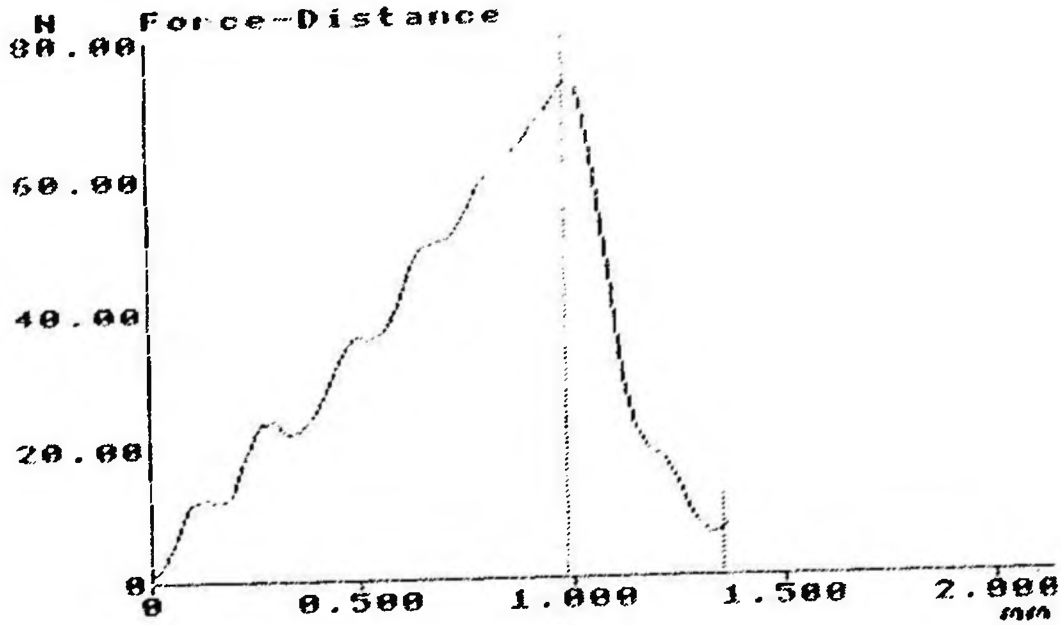
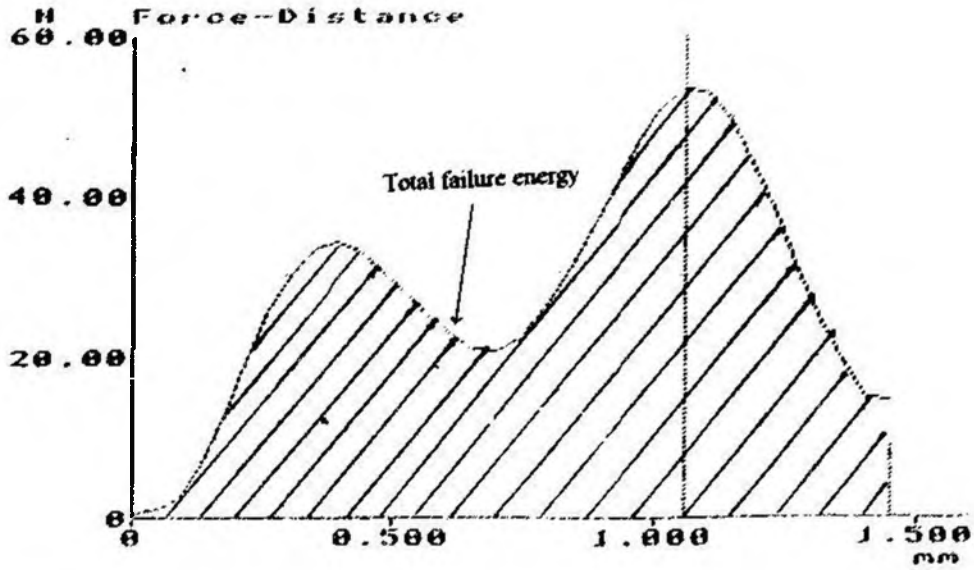


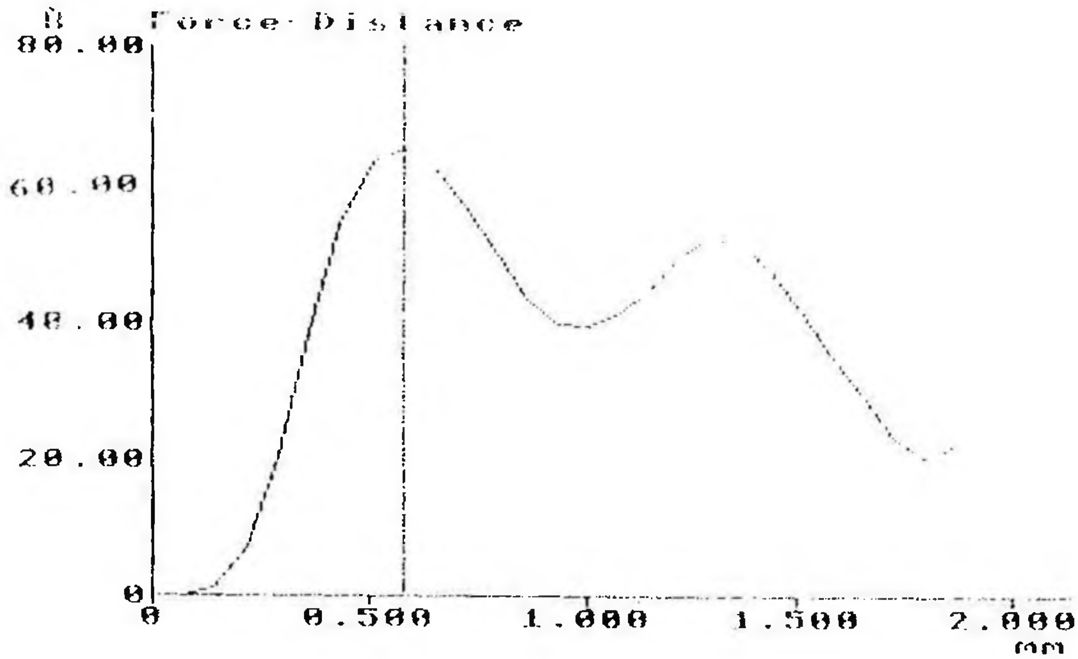
Figure 3: Effect of Impact Velocity on the Force Deflection Curve for Brittle Samples



(A) 1 m/s



(B) 3 m/s



(C) 4-6 m/s