

The Effect of Solvent Additions on the Mechanical Properties of Epoxy

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Abstract

The effect of the Toluene $C_6H_5CH_3$ and Acetone $(CH_3)_2CO$ additions on the mechanical properties of cured Epoxy is studied. Different weight percentages of Toluene (0, 4, 8, 10 & 14 wt%) and Acetone (0, 3, 5, 8 & 10 wt%) were used under curing temperatures (40°C and 60°C) for Toluene and 40°C for Acetone with curing time of 24hrs. The results show improved mechanical properties (Young modulus and Fracture stress) for Epoxy samples cured at 40°C with the increase of Toluene to a maximum of 8wt%, then these properties are degraded as the Toluene percentage increase. In the contrary, the Epoxy samples cured at 60°C shows poorer properties with increased weight percentages of the Toluene.

Epoxy samples thinned with different amount of Acetone solvent is prepared and cured. These samples show best mechanical properties at Acetone addition of 5wt%. Extra amounts of Acetone addition degrade the results. The mechanical properties, fracture morphology and FTIR spectra shows more aggressive effect of the Acetone compared with Toluene as solvent.

Keywords: polymer, solvent, mechanical properties, fracture morphology, FTIR spectra

تأثير اضافة المذيبات على المواصفات الميكانيكية للايبيوكسي الخلاصة

تمت دراسة تأثير اضافات التالوين $C_6H_5CH_3$ والاسيتون $(CH_3)_2CO$ على المواصفات الميكانيكية للايبيوكسي المعالج. وقد استخدمت نسب وزنية مختلفة من التالوين (0, 4, 8, 10 & 14 wt%) والاسيتون (0, 3, 5, 8 & 10 wt%) في درجتين حرارتين للمعالجة الحرارية للتالوين هما 40 و 60 درجة مئوية و 40 درجة مئوية للاسيتون ولمدة 24 ساعة. بينت النتائج تحسن المواصفات الميكانيكية (معامل يونك وجهد الكسر) للنماذج المعالجة بدرجة 40 مئوية مع زيادة نسبة التالوين حتى النسبة الوزنية 8% وبعدها بدأت تتراجع المواصفات مع زيادة نسبة التالوين. وعلى العكس من ذلك، ظهر ان النماذج المعالجة بدرجة حرارة 60 مئوية تعكس مواصفات ميكانيكية ضعيفة وتتراجع أكثر عند زيادة نسبة اضافة التالوين.

حضرت نماذج من الايبيوكسي مخففة بنسب مختلفة من الاسيتون. وكانت أفضل المواصفات الميكانيكية لهذه النماذج عند النسبة الوزنية 5% للاسيتون. وعند زيادة هذه النسبة تتراجع هذه النتائج. وقد بينت نتائج المواصفات الميكانيكية والصور المجهرية لسطح الكسر وكذلك فحوصات الأشعة تحت الحمراء تأثير أكثر حدة للاسيتون مقارنة التالوين كمذيب.

Introduction

The Epoxy is a well-known thermoset polymer for its dimensional stability, i.e. resistances to thermal and mechanical loading which is related to the degrees of cross linking or cross link density. It is familiar to decrease the viscosity Epoxy to facilitate handling and applying for various applications including crack healing and casting of a part. This is achieved by adding a given solvent to the buffer by a given extent and good mixing before adding the hardener. A recent work appeared on 2008 noticed that the fibers are easier for distribute in the resin after adding Acetone [1]. Thinning Epoxy with solvents led to the decrease in the viscosity of Epoxy which reduces the surface tension of the polymer-solvent mix [2]. Thus thinning Epoxy founds industrial attentions due to its role in improving adhesion, deeper penetration and else [3];

The dilution of polymer with solvent becomes an interesting experience outside the mechanical properties too; for example; it is shown that the electrical properties of polymer thin-film transistors PTFTs can be enhanced by controlling the solvent properties of solution used as the inkjet-printed source and drain electrodes [4]. A water-ethanol mix with different composition is also utilized as solvent for copolymer dilution too. It is shown that the solubility is varied according to water-ethanol combination [5].

Theoretically, it is shown that the orient-ability of the polymer chains is affected by the dilution and becomes more related to the flow during application which can affect its final mechanical properties [6, 7]. After the Epoxy has been set, it turns less soluble or ultimately insoluble

with solvents [8]. In addition, an interesting work appeared and explained a unique phenomena; that is; the presence of the solvent in the polymer matrix help for self healing of the micro-cracks after exposing to severe mechanical conditions. It is shown that solvent liquefy the interfaces which lead the Epoxy molecular chains to rejoin [9].

Modeling for the viscosity of polymer-solvent combinations has been explored and show that the viscosity of the system is related to the molecular structure [10]. The polymer chain, interacting with the solvent molecules through intermolecular forces is shown to vary the solvent conditions from those for good to poor solvents [11]. Another modeling effort shows that the extensibility (the maximum predicted length) of polymer chains plays an important role in predicting the macroscopic rheological behavior of dilute polymer solutions [12]. The rheological properties for dilute polymers are altered due to the presence of the solvent and thus, the elastic properties are enhanced as shown via theoretical calculations in a recent study [13].

The aim of this work is to presents experimental results for the mechanical properties, FTIR and the fracture morphology of Epoxy-Toluene system and Epoxy-Acetone system. In addition the results for the two systems are to be compared and discussed.

Experimental

Commercial type Epoxy resin (KUT Injectopox) has been used together with analar grade Toluene and Acetone as solvents. Different Epoxy-Toluene and Epoxy-Acetone percentages are prepared and tested for mechanical properties. Only the

Epoxy-solvent percentages that best reflect the behavior is presented to prevent overcrowding of the obtained data. Table 1 show the chosen Epoxy-Toluene and Epoxy-Acetone systems respectively.

The published standard tensile test method for plastics ASTM D638-03 [14] is used as guide for maintaining the shape and dimensions of the prepared samples for tensile tests. All samples were casted in carefully prepared silicon rubber molds.

The prepared Epoxy-solvent system has allowed for set for 24 hrs. Then, the samples are taken to oven for curing at 40°C for 24 hrs. An additional Epoxy-Toluene set (table 1) is prepared by the same procedure but cured at 60°C for 24 hrs to compare the tensile properties with that cured at 40°C.

A universal testing machine (Sanghai Mobis, China) is used to perform tensile tests for the cured samples with strain rate of 0.5mm/min. The resultant data are processed via an attached computer and stored as an excel charts. These charts are ready to process and extract useful property results.

In addition, an optical microscope (Bel, Italy) is used to observe the morphology of the failure zones. Micrographs of these zones are obtained with the aid of an attached camera to the microscope.

In order to explain the tensile test results; FTIR spectrographs are obtained for selected cured Epoxy samples (with and without solvents). The FTIR spectra are measure using shimadzu (USA) instrument in the range 400-4000cm⁻¹. However, for clear appearance of the shifts in the absorption bands; only the range of

800-1000cm⁻¹ is found useful and presented in this study.

Results and discussion

The stress-strain behavior of the Epoxy-Toluene system cured at 40°C for 24 hrs is shown in Figure (1). As, the Toluene percentage is increased up to 8 wt%, the tensile properties (namely young modulus and fracture stress) are improved. The results also show that for Toluene percentages more than 8 wt%, the tensile properties are decreased. These results can be explained in terms of that the Toluene solvent molecules impede or delay the cross-linking process by an extent proportional to the Toluene wt%. The net effect is reduction of the brittle character of the Epoxy and gradually developing ductility. In addition, the Toluene solvent is expected to improve the rheological properties of the polymer before setting as shown by other works theoretical works [10-13] which indicates better packing of the molecule chains. This is also explains the improvement of the young modulus at Toluene additions not exceeding 8wt%.

When the curing temperature was 60°C for 24 hrs, the fracture stress has its optimum value at 8 wt% again as shown in figure (2). It is expected a more aggressive action of Toluene solvent at 60°C; but it seems that action is not enough to break the molecular chains and reducing the strength at Toluene additions not exceeding 8wt%. On the contrary; the young modulus decreases at the Toluene percentage increases at all the range of Toluene wt%. These results indicate that the action of the Toluene solvent impedes cross linking of the polymer chains and reduces young modulus.

When the Toluene solvent is replaced by Acetone and the curing temperature is set to 40°C for 24hrs; the Acetone solvent shows similar behavior to Toluene as seen in figure (3). Both young modulus and fracture stress is improved as the Acetone percentage is increased, reaching an optimum values (3.389 GPa and 1.56 MPa respectively) then reduced at further increase of the Acetone percentages. The analysis of Epoxy-Acetone is the same for the Epoxy-Toluene system mentioned above except in the optimum solvent percentage. The optimum Acetone percentage was 5wt%, where it was 8wt% for the Toluene; i.e. a stronger effect of the Acetone solvent is realized.

Numerically; the young modulus and the fracture stress of the three mentioned Epoxy-solvent systems are shown in tables 2, 3 and 4. The data runs smoothly and monotonically and can draw smooth curves. Instead, these data is tabulated to permit easier utilization by a designer.

The morphology of the fracture surface is shown in figure (4) for the Epoxy-Toluene system. Part (a) of the figure shows mirror-like region that reflects typical (glassy) brittle fracture for amorphous polymer. At this region the fracture initiates due to localized chain slippage and reorientation of the molecule chain under the action of the stress. The molecule chains are stretched and the voids (dark areas) are expected to collapse and grow to the extent that allows the crack propagation. Therefore, the fracture area has a mirror-like character involving void areas. At some distance of the fracture initiation, the molecule chains have no enough time

for complete stretch. Thus, the region is expected to be a rougher region as shown in figure (4-b). This figure also reflects the dark areas of void coalescence. The in-betweens of the mirror-like and rough regions are shown in parts (c) and (d) of the figure.

Figure (5) presents the morphology of the fracture surface for Epoxy-Acetone system. Once more, the mirror-like region is shown in part (a), the rough region in part (b); In addition, in parts (c) and (d) shows the in-betweens regions. The main difference in the morphology of the fracture areas between that of the Epoxy-Toluene and Epoxy-Acetone systems is that larger void volumes appears. Furthermore, the whole regions appear rougher. These differences can be attributed to the aggressive nature of the Acetone that weakened the molecule chains and the failure starts before complete stretch. This led to observe rougher fracture morphology at whole fracture surface. The weaker molecule chains also expected to be responsible for the larger void volumes appeared in the morphology of the fracture surface.

The FTIR spectra are measured for four samples of the Epoxy solvent system as shown in figure (6). The first is for 0 wt% solvent, the second is for 8 wt% Toluene, the third for 14 wt% Toluene and the forth is for 5 wt% Acetone addition. In order to achieve better presentation, only limited range of the absorption spectra is presented. This range lies between 800-1000 cm^{-1} to focus on certain reference absorption band. This band is in the vicinity of 920 cm^{-1} which represent a characteristic absorption of Epoxy due to the contraction of the C-C bond and the stretching of C-O bond [1].

The reference band appears at 927.76cm^{-1} for the Epoxy without solvent addition; whereas, the band is shifted toward 931.62cm^{-1} and 945.12cm^{-1} for 8wt% and 14% Toluene addition respectively; i.e. increasing the solvent increases the numerical value of reference band location. In the cast of Acetone, the band shifted to 943.13cm^{-1} for 5wt% addition only. This once more led to the conclusion of that the Acetone addition has more aggressive effect on the structure of the cured polymer.

Conclusions

The solvent addition to the Epoxy matrix, to certain extent, improves the elastic properties by improving the ductility of the resultant cured polymer. The Acetone additions show more aggressive effect on the structure of the cured polymer compared with the Toluene.

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Table (1): The prepared Epoxy-Toluene system

Batch no.	wt% Toluene
T1	0
T2	4
T3	8
T4	10
T5	14
Batch no.	wt% Acetone
A1	0
A2	3
A3	5
A4	8
A5	10

Table (2): Tensile properties for Epoxy-Toluene system cured at 40°C

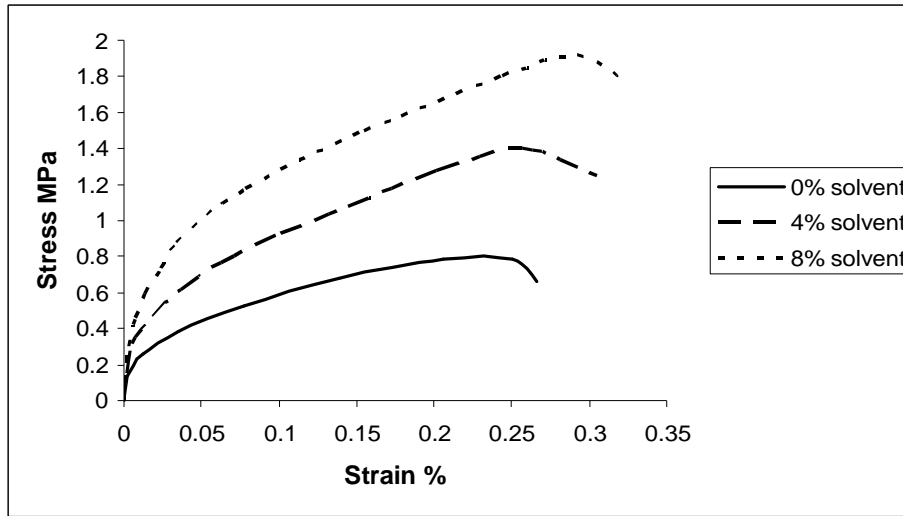
Sample no.	Toluene wt%	Young modulus GPa	Fracture stress MPa
1	0%	1.829	0.66
2	4%	2.664	1.09
3	8%	3.395	1.77
4	10%	3.238	1.39
5	14%	2.807	0.92

Table (3): Tensile properties for Epoxy-Toluene system cured at 60°C

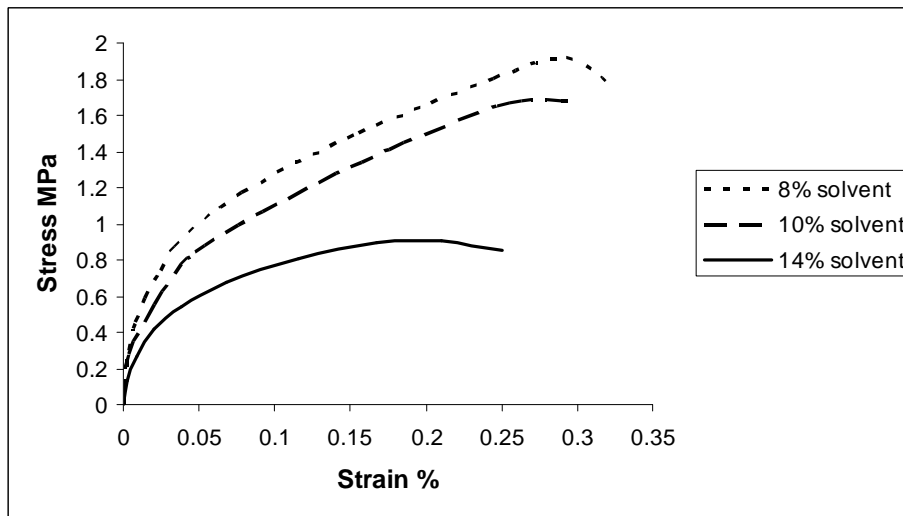
Sample no.	Toluene wt%	Young modulus GPa	Fracture stress MPa
1	0%	1.375	0.72
2	4%	0.632	0.92
3	8%	0.595	0.93
4	10%	0.589	0.73
5	14%	0.587	0.48

Table (4): Tensile properties for Epoxy-Acetone system cured at 40°C

Sample no.	Acetone wt%	Young modulus GPa	Fracture stress MPa
1	0%	1.829	0.66
2	2%	2.431	1.01
3	5%	3.389	1.56
4	8%	2.943	0.92
5	10%	2.369	0.83

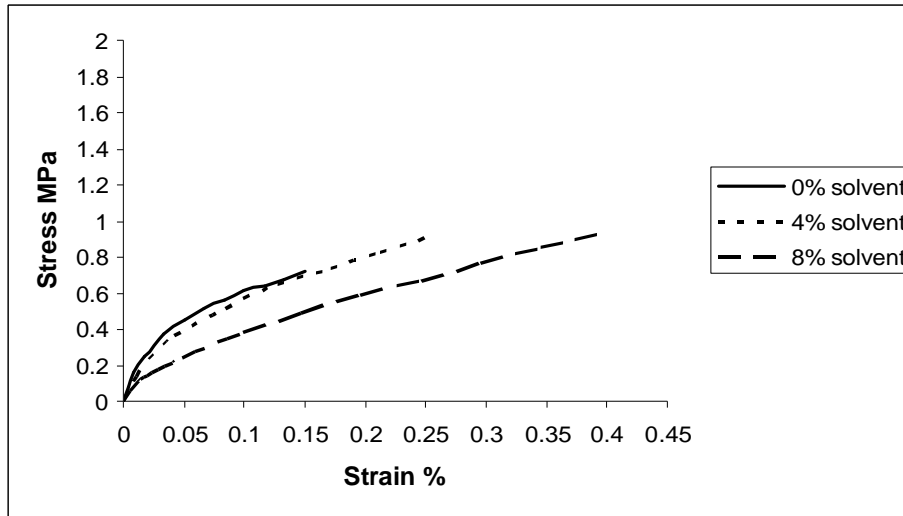


-a-

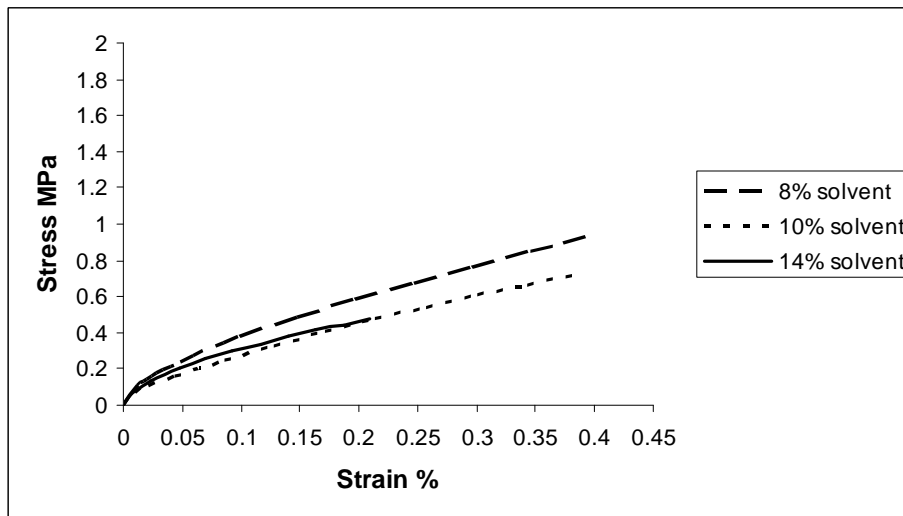


-b-

Figure (1) Tensile stress-strain behavior for the Epoxy-Toluene system showing best performance at Toluene solvent percentage of 8%. The samples are cured at 40°C for 24 hrs.

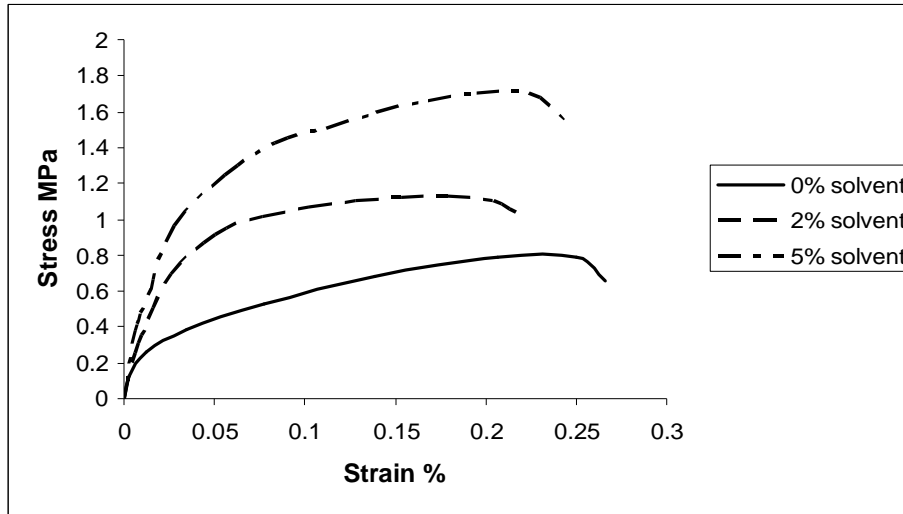


-a-

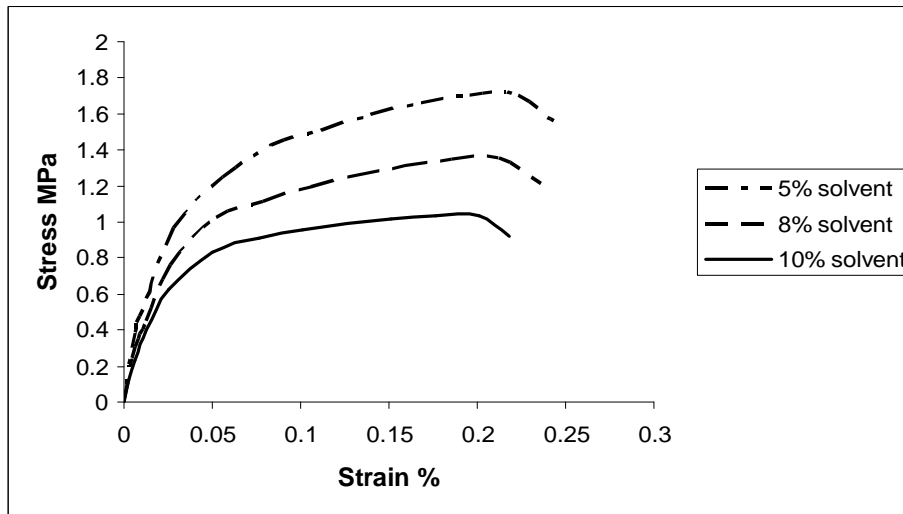


-b-

Figure (2) Tensile stress-strain behavior for the Epoxy-Toluene system cured at 60°C for 24 hrs showing reduced performance as the Toluene solvent percentage increased. The figure is splitted to (-a-) and (-b-) to ease comparing with figure (1) where the samples are cured at 40°C for 24 hrs.



-a-



-b-

Figure (3) Tensile stress-strain behavior for the Epoxy-Acetone system showing best performance at Acetone solvent percentage of 5%. The samples are cured at 40°C for 24 hrs.

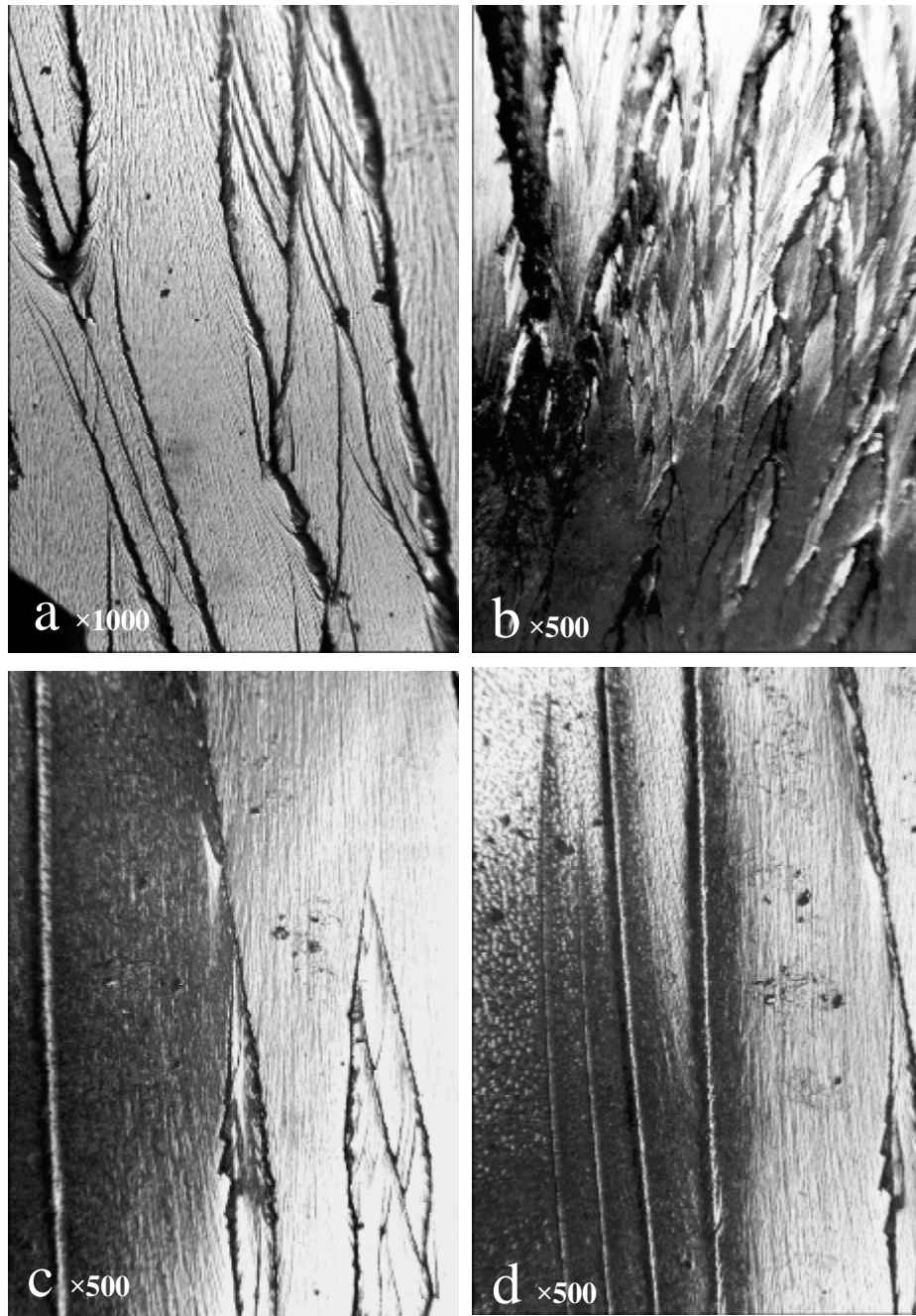


Figure (4) Surface morphology at different area of the fracture surface for Epoxy-Toluene system. The magnifications are shown at the figures. Figure (a) shows the brittle mirror like behavior at which the fracture starts; figure (b) shows rough fracture area apart from the crack initiation; figure (c) and (d) show transition regions from brittle to rough regions.

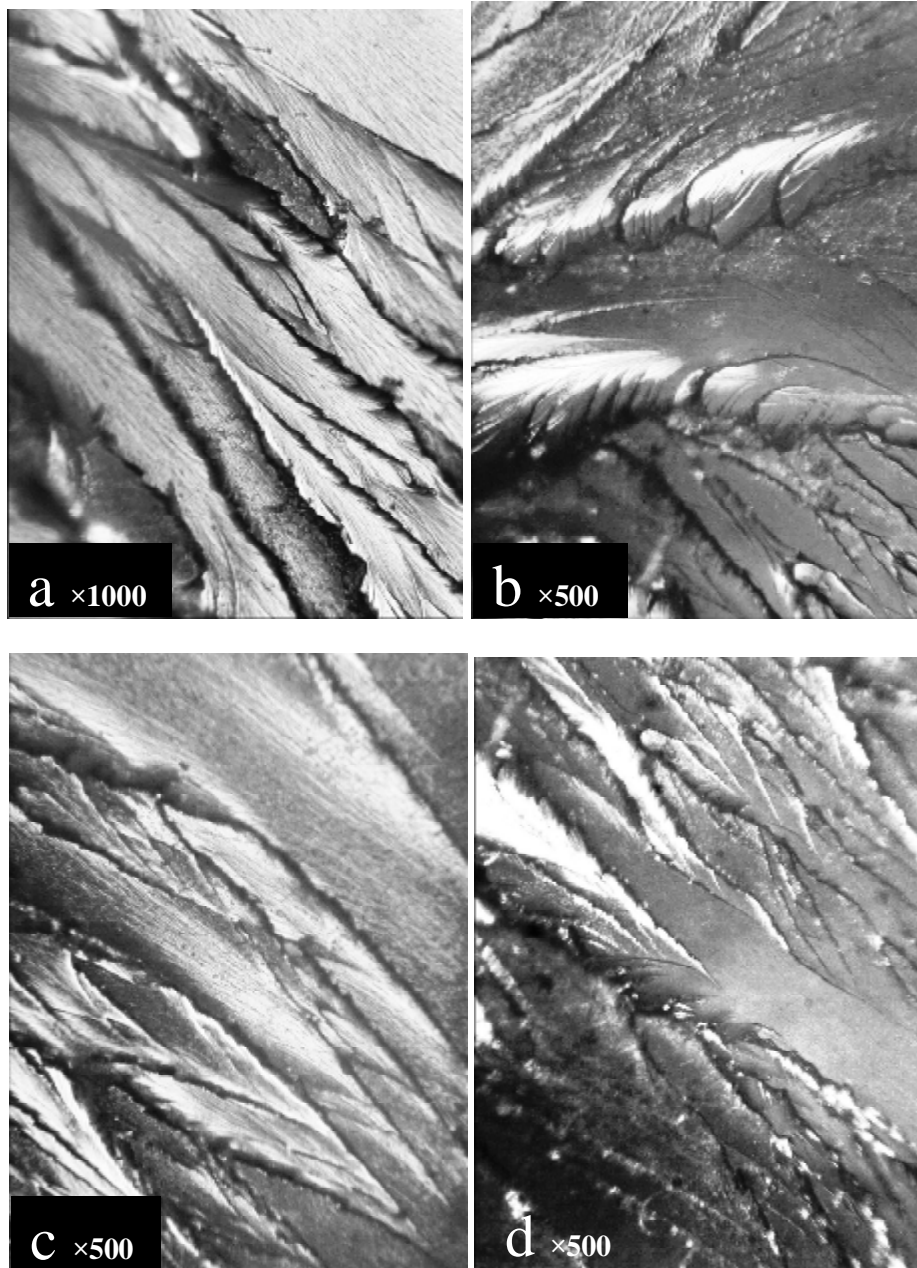


Figure (5) Surface morphology at different area of the fracture surface for Epoxy-Acetone system. The magnifications are shown at the figures. Figure (a) shows the brittle mirror like behavior at which the fracture starts; figure (b) shows rough fracture area apart from the crack initiation; figure (c) and (d) show transition regions from brittle to rough regions. The main difference from micrographs for the fracture surface of the Epoxy-Toluene system is the existence of larger voids

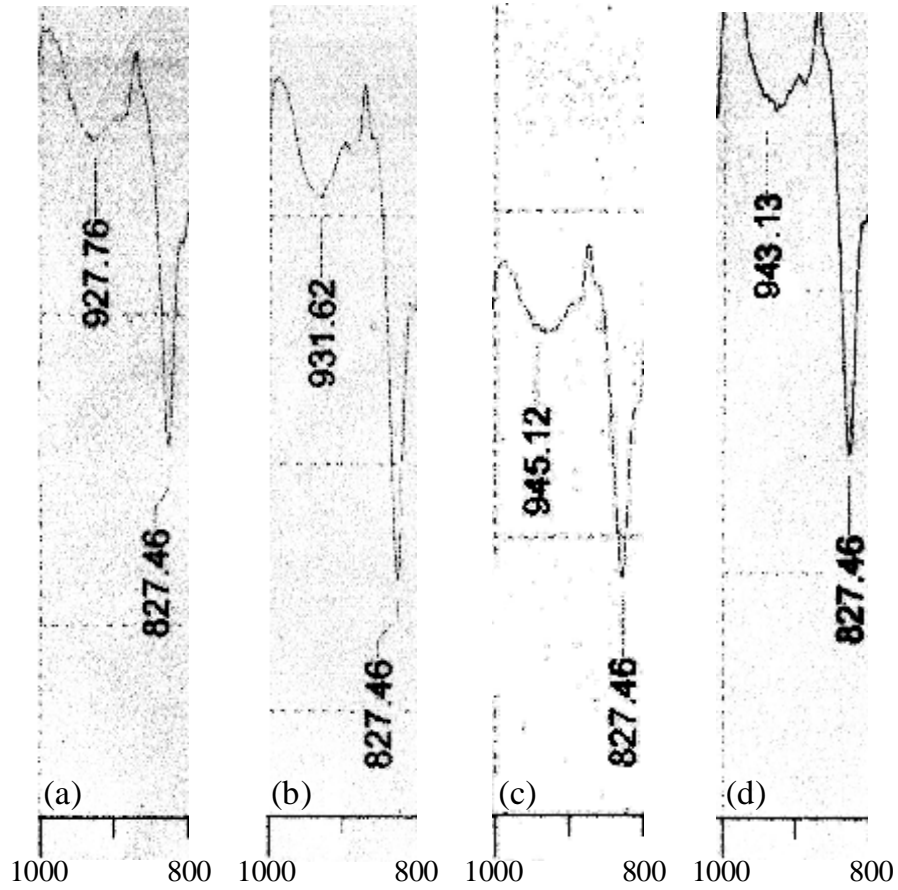


Figure (6) FTIR spectra for the Epoxy-solvent systems cropped to view the useful range of $800-1000\text{cm}^{-1}$ for clearer view. (a): 0wt% solvent, (b): 8wt% Toluene, (c): 14wt% Toluene and (d): 5wt% Acetone