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Effect of Various Process Variables on the Mechanical Properties of Laminates Produced through Resin Transfer Molding Process.

Sudheer Reddy Jarugu

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of
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for the Degree of Master of Applied Science at
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ABSTRACT

Effect of Various Process Variables on the Mechanical Properties of Laminates Produced through Resin Transfer Molding Process.

Sudheer Reddy Jarugu

The increasing interest in the Resin Transfer Molding Process (RTM) evolved from its potential for reducing manufacturing costs, while increasing the capability of manufacturing complex parts. This manufacturing process like many other processes involves a large number of variables, consisting of both material and processing parameters. The present study uses statistical experimental design analysis as a tool to determine which of the various independent and controllable RTM process variables affect the mechanical properties of the laminates produced through the RTM process. The statistical experiment is conducted in two phases. In the first phase, ten independent and controllable RTM process variables are chosen for study. For the first time, a sieve design is used to eliminate variables that are of not much importance. In the second phase, the important process variables are re-experimented, and the range at which they produce a good response (Laminate) is studied. In contrast to earlier studies, the present study considers the interaction effect of all the variables. Vacuum assistance is used during the mold filling, and its interaction affect on other factors, and effect on mechanical properties is also analyzed. In line with earlier studies, three different tests (mechanical three point bending, short beam shear, and dynamic mechanical test) are conducted to examine the possibility that a single important process variable might not have the same effect on all of them.

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Sudheer Reddy Jarugu

TO MY PARENTS.....

Sulochana...........Krishna Reddy

and...........Venkateswaruni Paada Padmamulaku.......

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CHAPTER 1

Resin Transfer Molding

1.1 Introduction.

Resin injection, or transfer molding (RTM), is a versatile and efficient process used in manufacturing of composite parts and structures. Although RTM has been in existence for decades, it is only of late that it has gained prominence in the composite industry. RTM process is gaining popularity due to its potential for forming near net-shape parts with a high tailorability of reinforcement, in a fairly repeatable manner. In addition RTM provides good surface quality on all sides, tight tolerances, tooling flexibility, short cycle times, and improved control of component shape, weight, and reinforcement volume fraction. A major attraction is the low capital investment related to this process as compared to similar processes such as filament winding and automated tape lay-up.

1.2 RTM Molding process

The elements of the process are shown schematically in Fig 1.0. Resin Transfer Molding is a relatively straight forward process that can be conceptually described in a few simple steps. A dry preform of reinforcing fiber materials is placed in a cavity in a mold half. The two halves of the mold are joined together and resin is injected into the mold, wetting the preform. The part is allowed to cure in the mold. It is then de-molded and finished.

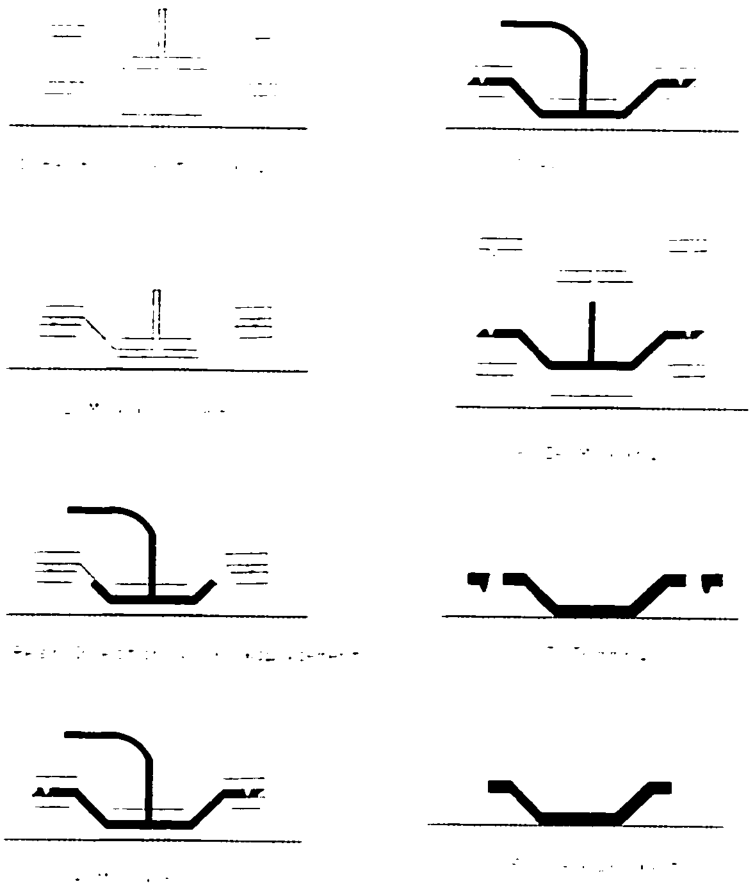

Fig 1.0 - Schematic diagram of the RTM cycle.

1.2 Variables of interest in RTM process.

RTM involves a large number of variables that are linked to the design of the component, the selection and formulation of constituent materials, and the design of the mold and molding process.

Many researchers have studied the RTM process in many different ways. A lot of research is being done to study the effects of both individual and coupled variables on the final composite part. Some of the important variables of RTM process where interest is shown include

1. Fiber architecture (materials).
    - Type of materials
        - ⇒ Continuous strand mat (Cosom).
        - ⇒ Plain weave.
        - ⇒ Knitted structure, e.t.c.,
    - Fiber fraction
        - ⇒ 20%
        - ⇒ 60%, e.t.c.,
2. Type of resin, resin flow in various fiber architectures, viscosity of resin, e.t.c.. (materials and processing).
3. Processing parameters (molding process)
    - Temperature of mold, resin & fiber.
    - Vacuum presence.
    - Curing temperature.
4. Machine parameters (molding process)
    - Injection pressure.
    - Shot length of the resin pump and additive pump.
    - Hydrocheck pressure.

- Type of shot (continuous, single).

5. Void formation during resin fill due to

    ⇒ Amount of mold releasing agent used on the mold.

    ⇒ Resin filling time.

    ⇒ Initial bubble content of the resin entering the mold during filling.

6. Void formation due to the effect of various processing & machine parameters.

1.3 Literature review

1.3.1 Introduction

Although RTM has been in existence for decades, it has recently gained prominence in the composites industry. Despite the general acknowledgment of the benefits of the RTM [1-7] and wide-spread industrial use, relatively little useful technical information appears to be available from which the designer can make appropriate choices of process, equipment, and material variables.

A lot of research is underway to understand the effects of the RTM variables, which were discussed earlier, on the composite laminates produced through this process [1,2,9,10]. Researchers are addressing the problems relating to the resin flow rates in various fiber architectures, its permeability, mold fill times, and the effect of these factors on the final structural performance. Some of the experiments are directed towards knowing the effects of various RTM variables on mechanical properties and void formation, which in turn affects the mechanical properties. The technology being naive little literature is available informing the contributions of various RTM variables for a quality laminate.

In the present context, literature relating to the studies which dealt with the effect of various variables on the mechanical properties and void formation in the laminates, is reviewed in detail. In line with the present scope of study, focus is laid on those topics where statistical experimental design analysis is used as a tool to ascertain the effect of various variables on the composite laminate produced through RTM process.

1.3.2. Effect of variables on Mechanical properties.

J.S. Hayward and B. Harris have studied the effects of process variables on RTM moldings along with vacuum assistance [1,2]. The variables that were under observation were : type of fiber architecture, resin type, variation in mold temperature, variation in injection pressure and, variation in resin viscosity. Very good results with vacuum assistance were reported by them, who found that the void volume fraction (V_v) was lowered considerably by the application of vacuum. Significant improvements were found in the mechanical properties and porosity levels of moldings made with vacuum assistance, compared with those produced without vacuum under otherwise identical conditions. Whatever the reinforcement type (whether woven, non-woven) or resin type and viscosity, vacuum assistance greatly improved the wetting of the fibers within a molding and thus the mechanical properties.

In to detail, the studies have shown a slight decrease in shear and flexural strengths with increasing injection pressure. With regard to the mold temperature (temperatures between 20-40 Deg.C.) the results indicated that molds heated to modest temperatures did not appear to affect the properties of RTM laminates in any way. The plates which were post cured for 24hours at 45 Deg.C. showed no significant changes, but the ones without post cure showed a decrease in flexural strength which was due to the different degrees of resin cure. In regard to the variations in resin viscosity (without vacuum), the studies have shown that selection of a suitable resin viscosity for a particular molding will be very dependent on the nature of the reinforcement, and in particular on the quantity of fiber in the mold (among other things). For example for a 40% fiber volume fraction, the high viscosity resin (~3500 mPas) produced a better part. Whereas when the volume fraction was 55% the low viscosity resin (~250mPas) produced a better part. So, as the volume fraction of fiber increases the higher viscosity resin cannot penetrate the reinforcement and wet out the fibers. Hayward and Harris conclude that the most likely reason for the improvement from vacuum assistance is that it is mainly a mechanical effect whereby the resin can penetrate more easily into voids when vacuum is used.

The effect of the preform architecture on resin flow, permeability and eventually on mechanical properties, was studied by Vistasp M.K, David A.S, and Giuiseppe R.P [13]. Three different glass fiber architectures were considered for investigation, i.e., Continuous strand mat (Cosom), a plain weave, and a (0/90) knitted structure respectively. Studies have shown that any change made in the fiber volume fraction and architecture affects the mechanical properties. Traditionally the transverse modulus and strengths should increase with an increase in the fiber weight fraction. However, studies have shown a decrease in the strength for an increase in the weight fraction. This trend is due to the increased compaction of preform layers, where fibers tend to crush, causing a decrease in the performance. This compaction can also affect permeability and flow, to an extent which is unexpected through just an increase in fiber weight fraction.

1.3.3. Void formation & process variables.

The problem of void formation in composites processing is very important since the physical and mechanical properties as well as the finish of the products are strongly influenced by the presence of voids. The examples of properties that are adversely affected by voids are the strength and the weather resistance of the composites. Several studies have reported a decrease of interlaminar shear and flexural strength with increase of the void content [14-16]. Other properties that may be diminished due to a high void content are, the dielectric strength and the surface finish of the composite [17-19].

The possible mechanisms for void formation and subsequent growth are numerous and are affected by, among others, injection pressure, suction pressure, temperature during injection and cure, pressure during cure, reinforcement characteristics (surface treatment, e.t.c), resin characteristics (surface tension, e.t.c.) and wetting angle between resin and reinforcement. Possible void formation mechanisms are mechanical entrapment of air already present in the mold and void growth after nucleation due to various mechanisms. The observations of J.S. Hayward and B. Harris [1,2], indicate that the main reason for void formation in RTM is mechanical entrapment rather than growth during cure which is an important source for voids in autoclave processed laminates [20].

The problem of void formation in RTM can roughly be seen on two scales. On a macroscale where the injection is viewed by the eye and on microscale where the flow between the fiber and tne fiber bundles is studied through a microscope. Many researchers including J.S. Hayward, B. Harris [1,2], T. Staffan Lundstorm, B. Rikard Gebart, C.Y. Lundemo [10,21], Wilson R. Stabler, Robert L. Sadler, Aly H.M. El-shiekh and Gary B. Tatterson [25], have addressed this problem and experimented to find out the causes of void formation using various process variables.

The effect from different processing parameters on the void content on a macroscale was investigated by Hayward and Harris [1,2], who pointed the importance of vacuum assistance during injection for the reduction of void content in the final part. When the vacuum on the outlet side of the mold repeatedly was turned on and off during processing a significant difference in transparency of the cured laminate was obtained. From this observation they suggest that the voids are formed at the resin flow front. The effect from vacuum assistance was further investigated by Lundstrom and Gebart [10,21], who studied the void content in rectangular plates produced with the RTM process. Parameters that are chosen for observation are as shown in Table 1.1. During the experimentation the resin was injected under constant pressure from one side of the mold to the other. The chosen injection strategy made the main flow unidirectional. Image analysis of micro graphs of the laminate shows that the voids are concentrated in an area near the outlet side of the laminate. Both this area and the void content in it decrease with the increase in vacuum level on the outlet side of the mold and they are practically zero at the highest vacuum level. Lundstrom and Gebart [21] extend the investigation to include several new processing parameters. In all cases the voids are found concentrated near the ventilation side of the laminate. The study has also shown that the reduction in void formation is directly proportional to the increase in the strength of the laminate.

The investigation also reveals that, besides vacuum assistance, the following processing conditions strongly reduce the void content of the finished part : high pressure during cure, resin flow out of the mold after complete filling and the right choice of resin and reinforcement. One interesting observation made was an increase in the void content with an increase in the processing temperature. Although the reason behind it was not investigated, they felt that with lower temperatures the resin viscosity is high and hence the reduction in void content. The summary of influences from processing parameters is as given below.

Table 1.1 - Summary of influences from processing parameters

Parameter	*Type of change*	*Average void content*	*Average length*	*Total void content*
Vacuum level	↑	↓	↓	↓
Injection length	↑	**Unclear**	↑	↑
Temperature of the mold	↑	↑	**No change**	↓
Injection pressure	↑	↑	↓	**Unclear**

In this study it is furthermore strongly indicated that the voids are generated at the resin flow front and that they move in the flow direction of the resin. The general explanation for the formation of bubbles at the resin flow front is that the permeability of the fibrous prefrom varies significantly on the micro level [22,23]. When resin is injected into fiber filled glass tubes [24] it is observed that relatively large bubbles(around 1mm in length) are trapped during injection. Some of the bubbles escape the entrapment and move along with the resin while others stay trapped between the fibers and decrease in size until they are not visible.

In their study, Wilson R. Stabler, Robert L. Sadler, Aly H.M. El-shiekh and Gary B. Tatterson [25], chose five processing variables : mold releasing agent, vibration frequency of the mold during filling, fill pressure, fill time and initial bubbles content in the resin, at different levels to establish the reasons for void formation. The study has shown that the excessive application of mold releasing agent produced more voids than light waxing followed by buffing. High initial bubble content has also increased the void formation. Vibration at 10 Hz reduced voids significantly in samples having high voidage and became less significant when near perfect samples were produced. The resin fill time and resin fill pressure has negligible effect on the void formation.

1.3.4 Use of Taguchi's design analysis to ascertain the effect of various process variables.

Dockum and Schell used the orthogonal array (L_8) of Taguchi in investigating a number of factors for the SRIM process [26]. However they restricted their study to the factors like : preform binder type, filament diameter, strand size, preform chop length and preform binder content. Their study does not focus on the critical issues related to microstructureprocessing property interactions except in a minor way, since no process related parameters were considered. In another related study, V.M. Karbhari, D.J. Wilkins , D.A. Steenkamer and S.G. Slotte, considered process related factors at two levels for examination [11]. The factors considered were, type of preform, number of preform layers, stroke length of the pumps, gating arrangement, injection pressure, shot type and tool temperature. The samples produced were tested for open hole tension and flexural properties. The contribution of each factor to the response was observed using the Taguchi's ANOVA. The analysis has given two different sets of parameters as being optimum for the open hole tension and the flexure tests, thereby signifying that the process and material parameters affecting each both type of mechanical behavior are different. The analysis has shown that the main contributors to the variation in results are the preform material type, the number of layers used, the type of gating, and the injection pressure. However, for flexure test results the stroke length appears to be the major contributor (77%) overshadowing all other factors. One interesting observation was that the standard deviation of the results noted was significantly lower indicating that the parameter set chosen was significantly insensitive to minor variations. The factors chosen for study and the resultant ANOVA are tabled in Tables 1.2,1.3,and1.4.

Table 1.2 - Variables and their Two levels

Factor	level 1	level 2
preform material type	A	B
Number of layers	3	5
Stroke length (inches)	3.5	6
Gating arrangement	Center	Corner
Injection pressure (psi)	40	75
Shot type	Single	Continuous
Tool Temperature	Room (70 Deg.F)	120 (Deg.F)

Table 1.3 - Analysis of the open-hole tests

Factor	Percent Contribution	Choice
Material Type	47.6	A
Number of layers	18.2	5
Stroke length (in)	2	3.5
Gating	11.5	Center
Injection pressure (psi)	16.4	40
Shot type	0.5	Continuous
Tool temperature	3.8	120

Table 1.4 - Analysis of the 4-point flexure tests.

Factor	Percent Contribution	Choice
Material Type	2	B
Number of layers	4	3
Stroke length (in)	77	3.5
Gating	4	Corner
Injection pressure (psi)	4	70
Shot type	3	Single
Tool temperature	5	Room (70 Deg.F)

1.4 Scope of the present work

In earlier studies of variables by J.S. Hayward et.al, only one parameter of interest is tested at a desired level whereas the other variables were held at the same level. This type of investigation would not aid in ascertaining the effect of various factors, since the behavior of the chosen variable is not tested at various levels of other factors to identify whether there is any change in the behavior of the factor. In the experimentation a little area was provided at the injection end, and at the suction end of the mold to study the flow pattern. However, in the real time manufacturing this might not be viable for many design and cost considerations. One more draw back in the study conducted by these authors is the consideration of non-independent factors, along with independent factors. One example is the resin viscosity. Resin viscosity is not an independent factor, since resin viscosity depends on the temperature. So either one of them should be considered for analysis. In this case authors considered both the factors. Some of the above problems were addressed in studies conducted by D.A. Steenkeenar et al[11], where they used Taguchi design analysis as a tool. The main drawback in Taguchi's design is that it doesnot address the interaction effects of the variables involved. However, ignoring interaction effects can result in a bias in the experiment if any such interactions are important, this can cause mis-estimation of the optimal setting of the control factors.

In the present study, experiments are conducted using the methodology of statistical design, wherein for the first time a sieve design is used [12]. Altogether ten independent and controllable variables are recognized for study, and are considered at two levels, a '+' level and a '-' level (various factors selected and their ranges are envisaged in Table 1.5). With the usage of sieve design in the first phase of the experiment one can eliminate the less significant variables, which will allow us to re-examine the significant variables, in the second phase, by constructing a full factorial statistical experimental design. This allows for a study of all possible processing variables and their interactions, with limited number of experiments. Also with the help of the new multiple regression model proposed by Al-Ata and Astakhov [27], instead of being content with the range at which there is an increase or decrease of response, this study will examine the values of variables at which the desired response (result) is maximum.

Table 1.5 - Factors chosen for experimentation.

Factors	Range '+' level	Range '-' level	Zero level	Units
Injection Pressure (A)	90	70	80	psi
Hydrocheck pressure(B)	80	60	70	psi
Mold temperature(C)	40	30	35	Deg.C
Post cure Temperature(D)	100	85	92.5	Deg.C
Valve open after filling (E)	YES	NO	-----	-----
Stroke length (F)	9	7	8	in.
Vacuum while molding (G)	10	5	7.5	in. Hg
No. Of layers (H)	5	4	----	Nos.
Type of reinforcement (I)	Chopped Matt.	COSOM	----	Type.
Resin to catalyst ratio (J)	100 : 1.5	100 : 2	100: 1.75	%

All the factors that are included in the study are as shown in Table 1.5. Even though some of the above mentioned factors were studied earlier, these factors are again considered since many new factors are added for observation. By leaving out some of the factors we might possibly miss their interaction effect which might have a significant effect on the result. The new factors that are not considered in the earlier studies include :

- Hydrocheck pressure: Hydrocheck pump is an extra fitting provided by the manufacturers of the RTM machine. The advantage of this pump is to provide a smooth effect of pumping the resin into the mold thereby reducing the bubbles, and hence the void formation.

- Valve position : The mold designed for the experiment consists of two ports, one for injecting the resin into mold, and the other to evacuate the mold using vacuum. In the case of the autoclave curing extra resin is squeezed out of the mold during the curing process, thus making the laminate fiber rich, and thus improving the mechanical properties. Study of this influence in RTM produced laminates would be an interesting observation. This is accomplished by varying the valve position, after injecting the resin, at two levels, open and closed, to ascertain whether it has any effect on the mechanical properties under study.

- Vacuum assistance: Studies have shown that vacuum assistance during molding has increased the mechanical properties of the laminates, by reducing the voids. Whether the vacuum presence will nullify the significance of machine variables would be an interesting paradigm to study. Experiments with higher injection pressures have also shown a release of the entrapped air. But a high pressure difference, i.e., a high resin flow rate, in the mold would cause in-sufficient wetting of fiber thus reducing the mechanical properties. In the first phase of the experiment it was decided to study the effect of higher injection pressures at different levels of vacuum, where in the pressure drop, in the mold was varied between 87.5, 85, 68.5, and 65 psi.

- Number of preform layers: Studies have shown a decrease of strength, for an increase in the number of layers or the weight fraction [13]. The reasons are the compaction of layers which is affecting the permeability and flow of resin causing improper wetting of the fibers. Whether the vacuum assistance has any influence is also examined by including the number of layers as one of the factors.

- Resin to Catalyst ratio : By a mechanism provided by the RTM machine manufacturer it is possible to vary the ratio of catalyst to resin. Catalyst in resin helps in providing a good cure, and cross linking thus improves the mechanical properties. At the same time, unused catalyst that is left out after the chemical reaction, aiding to decrease the mechanical properties. Hence this factor is included for study to ascertain its effect on the result.

Resin properties and resin flow are not included as a factor since they are non-independent factors. Resin viscosity changes with temperature (which is already considered as a factor), and also its flow varies with the number of layers (which is a factor).

Another issue to be addressed in this study is the type of mechanical testing. Prior analysis have shown that various process parameters have varying effect on different mechanical properties [11]. To ascertain this, three types of tests and five different mechanical properties are included for analysis.

One of the limitations of the present study is the usage of single response (one degree of freedom of response) to assess the affect of the factors.

CHAPTER 2

Experimental details

2.1 Introduction

The purpose of the present study is to identify the influence of various processing parameters on the final laminate produced through the RTM process. Some of the tools and materials used are as described below :

2.2 Tooling

The rectangular mold used in this study has three plates. The top plate is made up of Aluminum with dimensions of 14"X10"X2". A O-ring grove of 0.14" width and 0.083" depth is made on the base of the plate. Two holes of 0.25" diameter are made on the top side of the plate forming the inlet port and the outlet port.

The middle plate or the specimen window is made up of Aluminum (Al-6065) with window dimensions of 9"X5"X0.157", and the Aluminum plate dimensions are 14"X10"X0.157". The specimen laminate produced will be of 4mm or 0.157" thick.

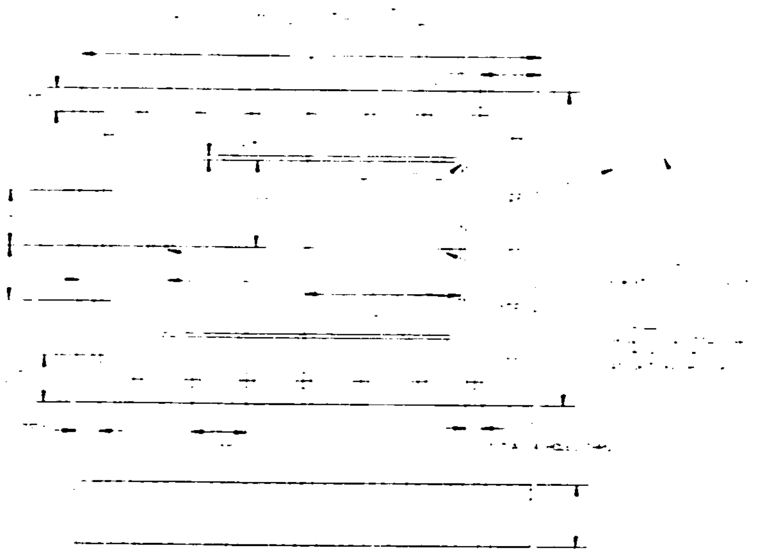

Fig. 2.1 - Aluminum (Top plate)

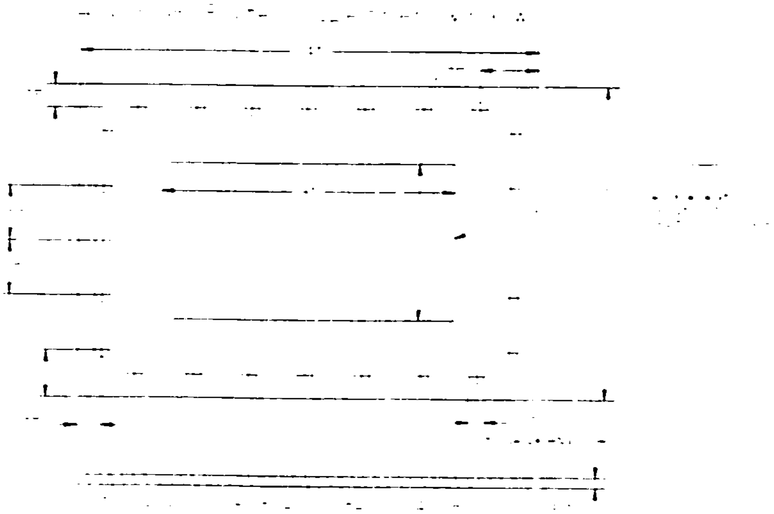

Fig. 2.2 - Aluminum plate (specimen window)

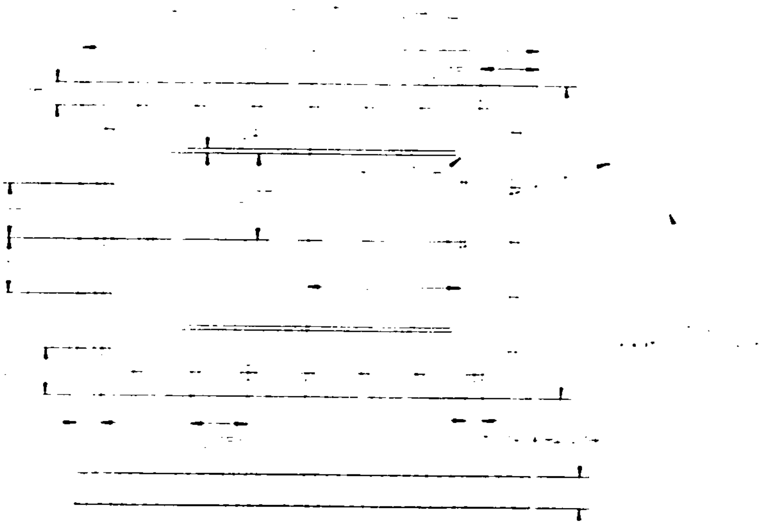

Fig. 2.3 - Aluminum plate (Base)

The bottom plate is also made up of Aluminum (Al-6065) with dimensions of 14"X10"X1". O-ring groves of 0.14" width and 0.03" depth are made on the top side of the base plate. Both O-ring groves help to seal the specimen window plate avoiding air leakage into resin when vacuum is applied. Twenty four holes of 0.5" through diameter are made along the sides of the mold facilitating the usage of bolts to clamp the mold.

Fig. 2.4 - Mold plates.

2.3 Re-inforcement

A continuous stranded mat (Cosom), and chopped stranded mat are used as reinforcements.

2.4 Resin, Catalyst and mold release agent

Resin used in the present study is Derakane-411-45, manufactured by Dow Chemicals, with a viscosity of less than 250 cps. The catalyst used for the present study is Cumene Hydroperoxide (CHP), and the mold releasing agent is a Release all-50.

2.5 Injection system

The injection system used in this study is a *Liquid Control Multiflow CVR*, variable ratio compact resin transfer molding machine, Model CMF4-5515 (fig 2.5). All the machine controls are pneumatically controlled under an air pressure of 90 psi. The machine is equipped with two pumps, one for the resin injection and the other one for the catalyst. Both the pumps called the Posiload positive-displacement metering pumps provides a high volumetric accuracy of +/- 0.5% from cycle to cycle. The posiload pump design allows for simple gravity feed of all self-leveling materials. The pump is capable of creating vacuum during its reload stroke, which assures complete metering tube filling without cavitation. The resin metering pump is connected to an additional hydraulic flow control device. This mechanism allows a rapid travel of the pump in the forward stroke, and then has a precision, adjustable constant speed throughout the metering portion of the cycle.

Fig. 2.5 - The Resin Transfer Molding Machine.

The Multiflow RTM machines offer variable resin to catalyst ratio's depending on the size of the metering pumps. This particular machine is fitted with a 10mm size catalyst metering pump, and a 55mm size resin metering pump, allowing resin to catalyst ratios from a maximum of 100 : 2.25 to a minimum of 100 : 0.7. The machine is also equipped with a shot-size selector for adjusting the length of the pump stroke.

The injection system is designed to allow the low volume catalyst to be injected into the center of the resin stream to assure a complete and uniform mixing in the Posimixer motionless mixer. Then the master bath (mixer of resin and catalyst) is injected into the mold from the end of the mixer nozzle.

2.6 Experimentation

The master bath is injected into the mold through the injection port. The outlet port is connected to a vacuum pump and vacuum is applied to remove the air cavities and reduce the void formation. The inlet port and outlet port are fitted with On-Off valves, made of Alloy 400. After complete filling of the mold the vacuum on the ventilation side was slowly released and the injection port was closed.

The entire mold assembly was maintained at a constant temperature during the injection of the resin into the mold. This is termed as the pre-mold temperature. This was accomplished with the help of a plastic tub of 12"X16" X5" dimensions. Brass rods were placed at the bottom of the tub and the mold was placed on the top of the brass rods, permitting for the water to flow underneath the mold. Water was added to the tub until the mold was completely immersed in the water. A heater with variable resistance was used to heat the water. The heater was provided with a revolving water jet which kept the water in constant motion providing uniform heat transfer. A thermocouple was attached to the mold to monitor the temperature of the mold.

The curing of the laminate after injection took place under atmospheric pressure in the mold, and the mold was transferred into an oven for post curing. The mold was allowed to stay in the oven for a period of 3 hours, and at a desired temperature, as specified by the resin manufacturer.

Fig. 2.6 - Experimentation set up for resin injection into the mold.

Statistical Experimental Design analysis demands identical environment during all the experimental runs. This was necessary to ensure precise assessment of the effect of various variables on the response. Some of the identical conditions maintained during the experiment are :

- Mold release agent was applied to the mold after every four specimens were manufactured.
- Care was taken while cutting the preform to its desired size.

- Reinforcement was placed in the mold prior to the heating of the mold and it was allowed to stay inside the mold until the temperature reaches a desired constant value.
- The mold was maintained at the desired temperature in the plastic tub with an accuracy of ± 1°C.
- Resin was not pre-heated before injection.
- Vacuum pump was turned on before the resin injection starts, and the mold was always evacuated before the injection of the resin.
- Resin was injected until the mold was completely filled, and the injection was stopped once there was an overflow of resin from the outlet port.
- Resin was allowed to post cure at the desired temperatures in an oven. Desired temperature was maintained inside the oven with the help of a thermostat fitted to the oven, with a accuracy of ± 1 °C. A thermocouple was used to cross check the temperature.

2.7 Specimens and Testing methods

The rectangular shaped laminate was cut into necessary specimen sizes depending upon the type of test and the required dimensions based on the ASTM standards. All the specimens were cut along the breadth, which acted as the length for the specimen. The specimens were cut for different tests randomly. This was to ensure that there was no bias in the specimen selection along the length of the laminate. The specimen dimensions for various tests are as shown in Table 2.1. Three different types of test are identified to study five different mechanical properties. A detailed description of the types of tests is given below.

Table 2.1 - Dimensions of the specimens used in three different tests.

Type of testing	Length (mm)	Width (mm)	Thickness (mm)	Span (mm)
Shear Strength	28	11	4	20
Flexural	100	25	4	65
DMA	**65**	**10**	**4**	**48**

2.7.1 3-Point Flexural tests

The testing method followed to determine flexural properties is as in ASTM - D 790M - 82. Among the two methods and procedures described in the standards, Method-I (3-point loading) and Procedure-B are followed depending upon the demands of the tests and materials. The flexural properties determined by this method are useful for quality control and specification purpose.

According to ASTM standards, specimens of high-strength reinforced composites, whose ratio of tensile strength to shear strength is less than 8:1, should have a span to thickness ratio of 16:1. For a laminate with 4 mm thickness the specimen sizes are as shown in Table 2.1.

2.7.2 Short beam shear test

The testing method followed to determine Interlaminar shear strength of fiber composites using the short beam test method, is as in ASTM-D 2344 - 84. Shear strength determined by this test method is useful for quality control and specification purposes, and it is also used to access the interply strength.

Standard size for a specimen made up of Glass fibers has a span to thickness ratio of 5, and length to thickness ratio of 7. For a laminate with a thickness of 4 mm the specimen sizes are as shown in Table 2.1.

An MTS machine (Fig 2.7) is used for conducting both the flexural and the shear tests. The speed of the cross head movement in the case of a flexural test is 2mm/min, and in the case of shear strength it is 1.3mm/min.

The formula used for calculating the flexural strength is:

$$F = 3PL / 2bd^2$$

where F = Flexural strength (MPA)

P = Load at failure (N)

L = Support span (mm)

b = Width of the specimen tested (mm)

d = Depth of the beam tested (mm)

The formula used for calculating the shear strength is :

$$S = 0.75\ P_b / bd$$

where S = Shear strength (MPA)

P_b = Breaking load (N)

b = Width of the specimen (mm)

d = Thickness of the specimen (mm)

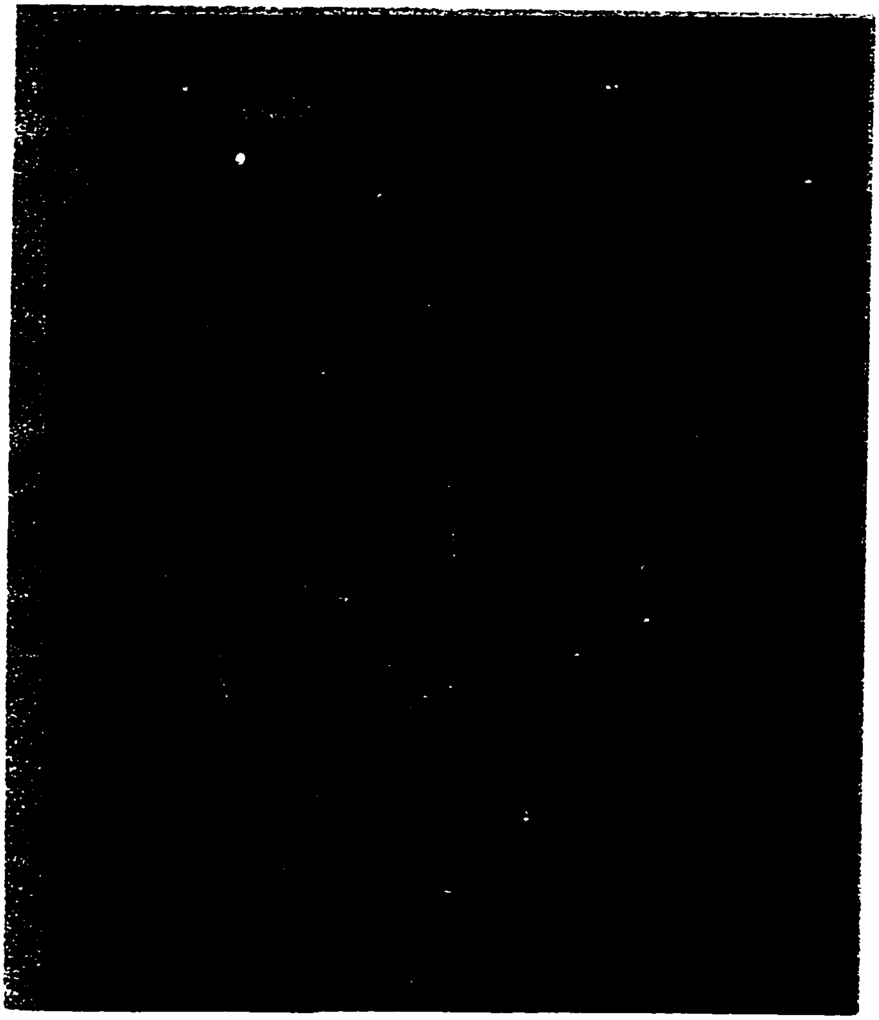

Fig. 2.7 - MTS machine used for testing flexural & shear properties.

2.7.3 Dynamic mechanical properties.

For studying flexural storage modulus, damping ratio and glass transition temperature a DMA-983 machine, manufactured by TA instruments is used (Fig 2.8).

Fig. 2.8 - Dynamic Mechanical Analyzer - DMA-983.

The values of storage modulus, damping ratio, and glass transition temperature are calculated by a built in computer program that accompanies the machine. Calculations involve a lot of machine parameters, and all the equations concerning these calculations are non-linear and undergo complex iterations to reach the final answer.

CHAPTER 3

Sieve Experiment : Methodology.

3.1 Introduction

The majority of the manufacturing processes contains a wide range of factors which have different affects (influence) on the final response (result). At the pre-process stage, an experimentalist wishes to include all essential factors into consideration. From one hand, a large number of factors makes experiment expensive and time consuming. On the other hand, when even one of essential factors is missed, the final model may be inadequate for the process under study. As such, no mistake at this stage should be made even though there is no ground to check it out before the final stage of the Design Of Experiments (DOE) where the data, collected in the experiments, are acquiesced and statistical criteria are examined. Therefore, the most important decision which has to be made at the pre-process stage is the decision about the factors to be included.

As one might expect, there are a few ways to deal with this problem. The first counts upon the experience of the experimentalist or research team. Since this way is the simplest, it is the most common. It is known from the ancient Greeks philosophy (Geradot, approx. 4 AD) that experience is good only to a certain extent and nothing else betray one's own experience. As shown by

Astakhov [12], experience adds a lot of different restrictions on a person which does not allow him or her to invent something new in spite of the existent experience. People are different so they have different background and experience. The correlation which one tries to establish experimentally is an objective one and exists regardless of what somebody thinks about it. Therefore, a main primary failure in such an approach is a subjective choice of the factors.

The second way is to take a fractional Design Of Experiments (DOE), which may dramatically reduce the number of necessary experiments. If it is believed that an interaction of second or higher order is not important, or that at the first stage of study the linear approximation is good enough, a fractional DOE may be used. A detailed analysis of such a kind of DOE [12] shows that detailed *priori* information about the chosen factor interaction is even more important than in the complete bloc DOE. Here, the efficiency depends to a large extent on a successful mixing of the linear effects with the interactions. The replicas (fractions of the complete block), where the linear effects are mixed with the interactions of higher orders, are the most effective and have the maximum contrast. An example is the use of 1/2048 fraction of the complete block 2^5 [12]. In this case, to do the complete block, 32768 tests have to be conducted. In contrast, the fractional DOE requires only 16 tests. However, for all these advantages the experimentalist pays a full price: all higher order interactions are excluded from the consideration. This is the price of simplicity.

By analysis of advantages and disadvantages of the first and second approaches, and by studying a large number of experimental results, Astakhov [12] proposed a new technique, sieve experiments, as a separate branch of DOE. Such a technique allows to include at the first stage of experimental study as many factors as the experimentalist wishes. Thereafter by conducting a relatively small number of the tests using special rules and statistical principles, the non-essential factors can be sieved out.

3.2 Methodology of The Sieve Experiment (SE).

Two different methods are used to construct the design matrix for SE [12]:

- In the first method the levels of factors are distributed within the matrix's columns by randomly using the table of random numbers;
- In the second method a SE matrix is accomplished by randomly mixing the two half-replicas taken from the corresponding complete block DOE.

The first method is considered as being less effective and is used when a number of the levels is greater than two. The second method looks more attractive because it involves only two levels of factor variation and a simple way to construct the design matrix.

The factors that are to be considered can be quantitative or qualitative but both types should be *controllable.* Practically this means that the chosen level of factor(s) are able to be set up and maintained with a certain accuracy within a test. The chosen factors should affect the process under investigation directly and not be functions of other factors. For example the resin viscosity cannot be chosen as a factor while temperature is included as a factor of study. Since, the resin viscosity depends upon the temperature. The factor's combinations should be compatible, i.e., all required factor's combinations should be realizable.

Each factor included in consideration has a certain range of variation. Within this range, the local sub-range which will be used in DOE has to be defined. Practically, it is necessary to define the limits of the each factor included in experiment. In order to do this, all available information can be used. It can be from experience, from results of previous study, etc. The choice of intervals of the factors variation is a non-formalized stage of DOE and is carried out according to the experience and intuition of the experimentalist. The accuracy of a factor setting at the chosen level, the degree of influence (correlation) of each factor on the response, and the accuracy of the response measurement should also be considered. Such considerations enable an experimentalist to avoid the situation when the chosen interval(s) of factor(s) variation is (are) not wide enough to detect the factor(s) influence on the response.

Summarizing the above consideration, it has to be pointed out here that even though at the stage of SE a lot of different factors may be included in consideration without a critical analysis of their influence on the response. But the controllability, accuracy of the measurement, maintenance of consistency within the experiment, independence, and interval of factor variation **must have a sufficient proof.**

In the present study SE is used to identify the significant factors affecting the RTM process. For the purpose of investigation ten independent, and controllable factors are selected and their intervals are decided based on prior studies. A complete list of all the factors and the intervals are shown in Table 3.1.

Table 3.1 - Parameters of Optimization (POO)

Factors	Range '+' level	Range '-' level	Zero level	Units
Injection Pressure (A)	90	70	80	psi
Hydrocheck Pressure(B)	80	60	70	psi
Mold Temperature(C)	40	30	35	Deg.C
Post Cure Temperature(D)	100	85	92.5	Deg.C
Valve open after filling (E)	YES	NO	-----	-----
Stroke Length (F)	9	7	8	in.
Vacuum while molding (G)	10	5	7.5	in. Hg
No. Of layers (H)	4	5	----	Nos.
Type of reinforcement (I)	Chopped Matt.	COSOM	----	Type.
Resin to Catalyst ratio (J)	100 : 1.5	100 : 2	100: 1.75	%

The design matrix for the SE, for the present study has been built as follows. All the chosen factors are divided into two groups. The first one contain factors A, B, C, D, and E which form a half-replica 2^{5-1}, with decisive contrast i = ABCDE. In this half-replica the effects of factors and the effects of interactions are not mixed. The remaining factors F,G,H,I and J are used to form the next half replica in the same way as the first one. Both the half replicas with their decisive contrast are shown in Tables 3.2, 3.3 and 3.4, 3.5 respectively. The required SE design matrix is then formed from the naturally written first half-replica and by adding after its each row, the row randomly chosen from the second replica. Here, randomization of the second replica's rows is very important and has to be accomplished using a table or a generator of random numbers. Formed by this way, the full SE design matrix is shown in Table 3.6.

As soon as the design matrix is completed, the check out of its suitability has to be done. A design matrix is called a suitable matrix if it does not contain two identical columns (with the same or alternative signs). Moreover, the matrix should not contain columns which scalar product appears as a column with an (+) or a (-).

To analyze the results of a sieve experiment Astakhov [12], has proposed the so called "*scatter diagrams*". *By* definition, the scatter diagram is a diagram in xy-coordinates where the chosen factors with their levels are mapped along the *x-axis* and the responses corresponding to these factors are pointed along y-axis. Each factor is considered individually, i.e., without any connection with others. Significant linear effects may be distinguished by direct observation.

Table 3.2 - Design Matrix of the first half replica

Runs	*A*	*B*	*C*	*D*	*E*
i	-	-	-	-	-
A	+	-	-	-	-
B	-	+	-	-	-
AB	+	+	-	-	-
C	-	-	+	-	-
AC	+	-	+	-	-
BC	-	+	+	-	-
ABC	+	+	+	-	-
D	-	-	-	+	-
AD	+	-	-	+	-
BD	-	+	-	+	-
ABD	+	+	-	+	-
CD	-	-	+	+	-
ACD	+	-	+	+	-
BCD	-	+	+	+	-
ABCD	+	+	+	+	-
E	-	-	-	-	+
AE	+	-	-	-	+
BE	-	+	-	-	+
ABE	+	+	-	-	+
CE	-	-	+	-	+
ACE	+	-	+	-	+
BCE	-	+	+	-	+
ABCE	+	+	+	-	+
DE	-	-	-	+	+
ADE	+	-	-	+	+
BDE	-	+	-	+	+
ABDE	+	+	-	+	+
CDE	-	-	+	+	+
ACDE	+	-	+	+	+
BCDE	-	+	+	+	+
ABCDE	+	+	+	+	+

Table 3.3 - Design Matrix of the second half replica

Runs	F	G	H	I	J
I	-	-	-	-	-
F	+	-	-	-	-
G	-	+	-	-	-
FG	+	+	-	-	-
H	-	-	+	-	-
FH	+	-	+	-	-
GH	-	+	+	-	-
FGH	+	+	+	-	-
I	-	-	-	+	-
FI	+	-	-	+	-
GI	-	+	-	+	-
FGI	+	+	-	+	-
HI	-	-	+	+	-
FHI	+	-	+	+	-
GHI	-	+	+	+	-
FGHI	+	+	+	+	-
J	-	-	-	-	+
FJ	+	-	-	-	+
GJ	-	+	-	-	+
FGJ	+	+	-	-	+
HJ	-	-	+	-	+
FHJ	+	-	+	-	+
GHJ	-	+	+	-	+
FGHJ	+	+	+	-	+
IJ	-	-	-	+	+
FIJ	+	-	-	+	+
GIJ	-	+	-	+	+
FGIJ	+	+	-	+	+
HIJ	-	-	+	+	+
FHIJ	+	-	+	+	+
GHIJ	-	+	+	+	+
FGHIJ	+	+	+	+	+

Table 3.4 - The decisive contrast for the first half replica : i = ABCDE.

1	A	BCDE
2	B	ACDE
3	C	ABDE
4	D	ABCE
5	E	ABCD
6	AB	CDE
7	AC	BDE
8	AD	BCE
9	AE	BCD
10	BC	ADE
11	BD	ACE
12	BE	ACD
13	CD	ABE
14	CE	ABD
15	DE	ABC
16	ABCDE	i

Table 3.5 - The decisive contrast for the second replica : i = FGHIJ.

1	F	GHIJ
2	G	FHIJ
3	H	FGIJ
4	I	FGHJ
5	J	FGHI
6	FG	HIJ
7	FH	GIJ
8	FI	GHJ
9	FJ	GHI
10	GH	FIJ
11	GI	FHJ
12	GJ	FHI
13	HI	FGJ
14	HJ	FGI
15	IJ	FGH
16	FGHIJ	i

Table 3.6 - Randomly selected design matrix

Runs.	Factors									
NO'S	A	B	C	D	E	F	G	H	I	J
1	+	-	-	-	-	+	-	-	-	+
2	-	+	-	-	-	-	+	-	-	+
3	-	-	+	-	-	-	-	-	-	+
4	-	-	-	+	-	-	-	-	+	-
5	-	-	-	-	+	-	-	-	+	+
6	+	+	-	-	-	+	-	-	-	-
7	+	-	+	-	-	+	-	+	-	-
8	+	-	-	+	-	-	-	+	+	-
9	+	-	-	-	+	-	+	-	+	-
10	-	+	+	-	-	-	-	+	-	-
11	-	+	-	+	-	-	+	-	-	-
12	-	+	-	-	+	-	-	+	-	+
13	-	-	+	+	-	-	+	+	-	+
14	-	-	+	-	+	-	-	+	-	+
15	-	-	-	+	+	+	+	-	-	-
16	+	+	+	+	+	+	+	+	+	+

The scatter diagram for the main factor analysis of glass transition temperature is shown in Fig. 3.1. Here, the linear effects are shown by horizontal axis. The number of points distinguished in the upper and lower parts of the scatter diagram may be also considered. For example, consider the factor *D* (Fig. 3.1). There are six points at the "+" level for which values of the response y are bigger than the biggest response at the (-)level. Furthermore, there are ten points at '-' level for which the responses are lower than the lowest response at the (+) level. Therefore, the total number of the distinguished points is equal to sixteen for the factor *D*. The large number of distinguished points means that the effect of the factor is strong. In the present case, at the first

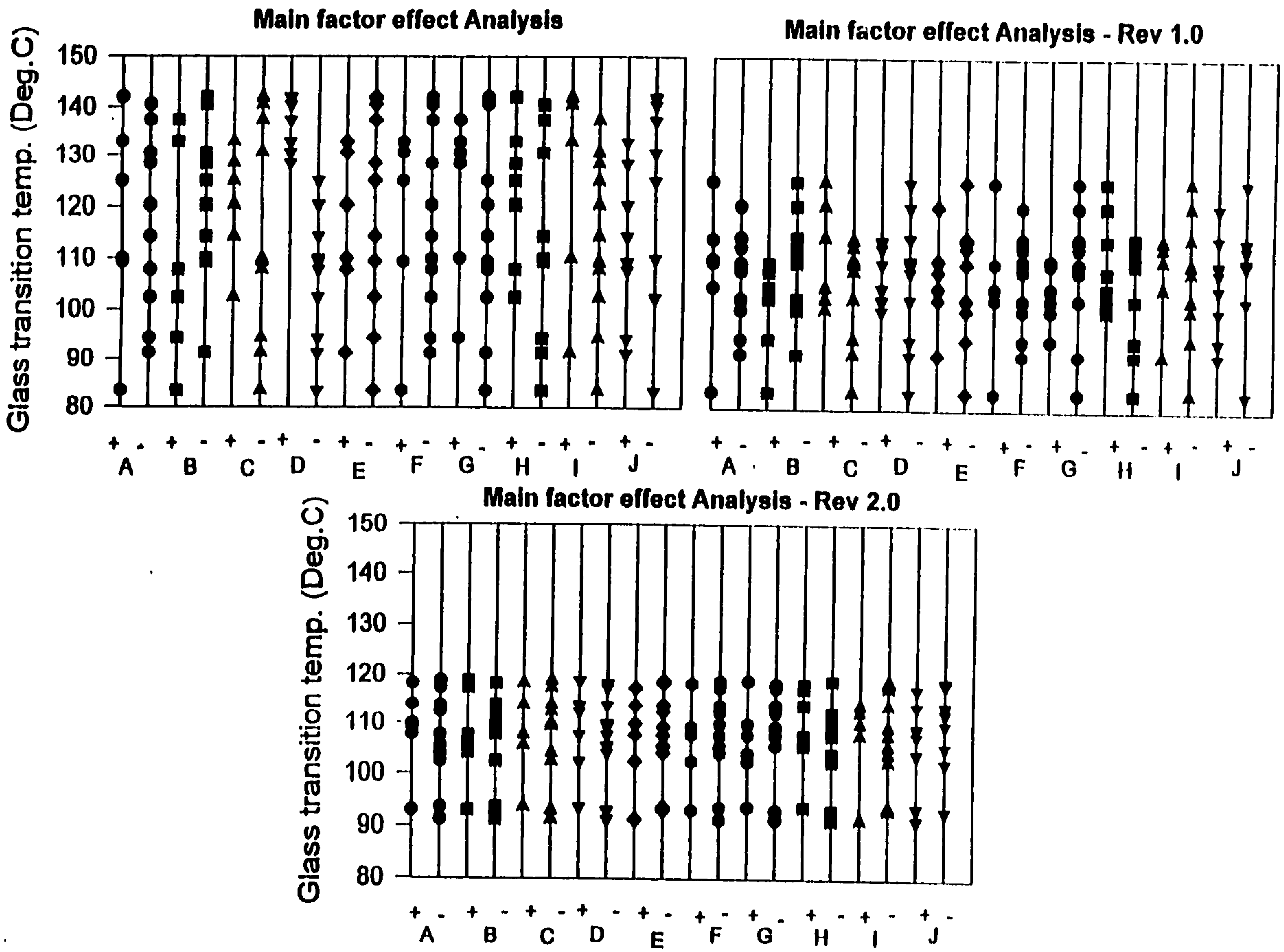

Fig. 3.1 - Glass transition temperature main factor analysis.

stage of study, factors D and H were recognized to have linear effects. The factors' effects are estimated quantitatively using tables with a few inputs. The table with two inputs is constructed for the considered case (Table 3.8).

Table 3.7 contains the experimental results obtained using the different combinations of the factors' levels. The value of an effect is calculated as

$$S = \sqrt{\frac{\sum y_i^2}{n_i - 1} - \frac{\sum (y_i)^2}{n_i(n_i - 1)}} \qquad \text{std} = \sqrt{\sum \frac{s^2}{n_i}}$$

Where 'std' is the standard deviation.

Where 'n_i' is the number of terms at the particular level of C and D factors.

Table 3.7 - Results of Glass transition temperature at various runs.

Runs	Factors											
NO'S	A	B	C	D	E	F	G	H	I	J	Average	Sieve 1
1	+	-	-	-	-	+	-	-	-	+	109.22	109.22
2	-	+	-	-	-	-	+	-	-	+	94.237	94.237
3	-	-	+	-	-	-	-	-	-	+	114.15	114.15
4	-	-	-	+	-	-	-	-	+	-	140.48	113.73
5	-	-	-	-	+	-	-	-	+	+	91.139	91.139
6	+	+	-	-	-	+	-	-	-	-	83.252	83.252
7	+	-	+	-	-	+	-	+	-	-	124.79	124.79
8	+	-	-	+	-	-	-	+	+	-	141.93	115.18
9	+	-	-	-	+	-	+	-	+	-	109.84	109.84
10	-	+	+	-	-	-	-	+	-	-	102.29	102.29
11	-	+	-	+	-	-	+	-	-	-	137.34	110.59
12	-	+	-	-	+	-	-	+	-	+	107.66	107.66
13	-	-	+	+	-	-	+	+	-	+	128.30	101.55
14	-	-	+	-	+	-	-	+	-	+	120.18	120.18
15	-	-	-	+	+	+	+	-	-	-	130.44	103.69
16	+	+	+	+	+	+	+	+	+	+	132.75	106.00

Table 3.8 - Calculation of main effects for Glass Transition Temperature

	+D		-D	
y_i	+H	-H	+H	-H
	141.9287	140.4787	124.7857	109.218
	128.2977	137.336	102.287	94.2366
	132.752	130.4373	107.6633	114.1487
			120.1793	91.1392
				83.2516
				109.8433
	y_1	y_2	y_3	y_4
Σy_i	402.9784	408.252	454.9153	601.8374
Mean of y_i	134.3261	136.084	113.7288	100.3064
$(\Sigma y_i)2$	1.6239E+5	1.6667E+5	2.0395E+5	3.6221E+5
$(y_i)^2$	5.4227E+4	5.5609E+4	5.2069E+4	6.1142E+4
n_i	3	3	4	6
s^2	48.3095	26.3831	110.523	154.7487
s^2/n_i	16.1032	8.7944	27.6308	25.7915
Std.	8.8498			

$$\text{Effect of D} = \frac{\bar{y}_1+\bar{y}_3}{2}-\frac{\bar{y}_2+\bar{y}_4}{2} = \frac{134.3261+113.7288}{2}-\frac{136.084+100.3064}{2} = 28.1875$$

$$\text{Effect of H} = \frac{\bar{y}_1+\bar{y}_2}{2}-\frac{\bar{y}_3+\bar{y}_4}{2}$$

= [(134.3261+136.084)/2 - 113.7288+100.3064)/2] = 5.8323

$$\text{t-significance of D} = \frac{(\bar{y}1+\bar{y}3)-(\bar{y}2+\bar{y}4)}{\text{std}}$$

= [(134.3261+113.7288)/8.8498 - (136.084+100.3064)/8.8498] = 6.3702

$$\text{t-significance of C} = \frac{(\bar{y1} + \bar{y2}) - (\bar{y3} + \bar{y4})}{\text{std}}$$

$$= [(134.3261+136.084)/8.8498 - (113.7288+100.3064)/8.8498] = 1.3181$$

Degrees of freedom $f=\sum n_i - k$ (where k is the number of cells, i.e., 4)=12.

From the t-distribution table [27], for 12 degrees of freedom $t_{0.10} = 1.35$. Since effect of factor 'D' is above '1.35', it is then considered to be significant with a probability of 95%. The effect of factor 'H' being less than '1.35', it is considered insignificant.

After the estimation of strong effects, the correction of the experimental result should be made to distinguish less strong effects and interactions. This correction is made by adding the (with reverse signs) distinguished effects to the results of the original sieve experiment, thus -26.75 is added to all results where the level of 'D' was '+'. If factor 'C' was also a distinguished factor then the same above procedure is repeated. The results after the correction are shown in Table 3.8, column labeled as sieve1.

Using the corrected results, a new scatter diagram is constructed as shown in Fig 3.1 (main factor analysis - Rev 1.0). Again the same procedure of calculating the factors' estimations and the corrections of the results are conducted as long as the factors' effect become insignificant for 10% level of significance.

When all corrections of the experimental results are over, the diagram of the response distribution at the passed stages of correction is constructed in Fig 3.1, (main factor analysis - Rev 2.0). As one might expect, the scatter of the experimental points becomes smaller with each stage of correction.

3.2.1 Factor interaction analysis.

In the present sieve analysis only two factor interaction is considered. The higher factor interactions are neglected. Since the objective of the analysis is to eliminate the insignificant factors, a two factor interaction can give an initial idea of the interaction effect, which is sufficient to judge the significance of the factor.

The necessary design matrix for the interaction analysis is as shown in Table 3.9. This design matrix is obtained by multiplying the level of one factor, at a particular run, with the level of another interaction factor. So a '+', and a '+', will yield a '+', and a '-', and '+', will yield a '-'. The necessary calculations, for estimation of effects, and significance of the interactions, are as explained in the above section.

Table 3.9 - Design matrix for factor interaction analysis.

Run	Interaction factors																					
NO'S	A B	A C	A D	A E	A F	A G	A H	A I	J A	B C	B D	B E	B F	B G	B H	B I	B J	C D	C E	C F	C G	C H
1	-	-	-	-	+	-	-	-	+	+	+	+	-	+	+	+	-	+	+	-	+	+
2	-	+	+	+	+	-	+	+	-	-	-	-	-	+	-	-	+	+	+	+	-	+
3	+	-	+	+	+	+	+	+	-	-	+	+	+	+	+	+	-	-	-	-	-	-
4	+	+	-	+	+	+	+	-	+	+	-	+	+	+	+	-	+	-	+	+	+	+
5	+	+	+	-	+	+	+	-	-	+	+	-	+	+	+	-	-	+	-	+	+	+
6	+	-	-	-	+	-	-	-	-	-	-	-	+	-	-	-	-	+	+	-	+	+
7	-	+	-	-	+	-	+	-	-	-	+	+	-	+	-	+	+	-	-	+	-	+
8	-	-	+	-	-	-	+	+	-	+	-	+	+	+	-	-	+	-	+	+	+	-
9	-	-	-	+	-	+	-	+	-	+	+	-	+	-	+	-	+	+	-	+	-	+
10	-	-	+	+	+	+	-	+	+	+	-	-	-	-	+	-	-	-	-	-	-	+
11	-	+	-	+	+	-	+	+	+	-	+	-	-	+	-	-	-	-	+	+	-	+
12	-	+	+	-	+	+	-	+	-	-	-	+	-	-	+	-	+	+	-	+	+	-
13	+	-	-	+	+	-	-	+	-	-	-	+	+	-	-	+	-	+	-	-	+	+
14	+	-	+	-	+	+	-	+	-	-	+	-	+	+	-	+	-	-	+	-	-	+
15	+	+	-	-	-	-	+	+	+	+	-	-	-	-	+	+	+	-	-	-	-	+
16	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+

Run	Interaction factors																					
NO'S	C I	C J	D E	D F	D G	D H	D J	E F	E G	E H	E I	E J	F G	F H	F I	F J	G H	G I	G J	H I	H J	I J
1	+	-	+	-	+	+	-	-	+	+	+	-	-	-	-	+	+	+	-	+	-	-
2	+	-	+	+	-	+	-	+	-	+	+	-	-	+	+	-	-	-	+	+	-	-
3	-	+	+	+	+	+	-	+	+	+	+	-	+	+	+	-	+	+	-	+	-	-
4	-	+	-	-	-	-	-	+	+	+	-	+	+	+	-	+	+	-	+	-	+	-
5	-	-	-	+	+	+	-	-	-	-	+	+	+	+	-	-	+	-	-	-	-	+
6	+	+	+	-	+	+	+	-	+	+	+	+	-	-	-	-	+	+	+	+	+	+
7	-	-	+	-	+	-	+	-	+	-	+	+	-	+	-	-	-	+	+	-	-	+
8	-	+	-	-	-	+	-	+	+	-	-	+	+	-	-	+	-	-	+	+	-	-
9	-	+	-	+	-	+	+	-	+	-	+	-	-	+	-	+	-	+	-	-	+	-
10	-	-	+	+	+	-	+	+	+	-	+	+	+	-	+	+	-	+	+	-	-	+
11	+	+	-	-	+	-	-	+	-	+	+	+	-	+	+	+	-	-	-	+	+	+
12	+	-	-	+	+	-	-	-	-	+	-	+	+	-	+	-	-	+	-	-	+	-
13	-	+	-	-	+	+	+	+	-	-	+	-	-	-	+	-	+	-	+	-	+	-
14	-	+	-	+	+	-	-	-	-	+	-	+	+	-	+	-	-	+	-	-	+	-
15	+	+	+	+	+	-	-	+	+	-	-	-	+	-	-	-	-	-	-	+	+	+
16	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+

3.3 Analysis of the sieve experimental results.

Sieve design analysis is used to ascertain the significant factors among various RTM process parameters. Five mechanical properties are studied to investigate the effect of the factors on each of the mechanical properties. Both the main and interaction effects of the factors, on all the mechanical properties under study, are analyzed using the scatter diagrams. All the scatter diagrams obtained in the sieve analysis are shown in appendix (A). All the calculations obtained in the sieve analysis are shown in appendix (B). All the graphs obtained for the five mechanical properties are appended as Appendix (C).

Table 3.10 - Results of five mechanical properties.

Run	Shear Strength (MPa)	Flexural Strength (MPa)	Damping ratio	Flexural storage Modulus (GPa)	Glass transition temp. (Deg.C)
1	24.10	220.00	0.2225	6.9854	109.218
2	23.60	206.00	0.2024	6.7694	94.2366
3	24.80	209.00	0.2239	6.6992	114.1487
4	26.10	176.90	0.2312	7.7316	140.4787
5	16.00	179.00	0.2063	7.0880	91.1392
6	18.70	143.00	0.1680	8.7336	83.2516
7	25.90	214.50	0.2090	7.4504	124.7857
8	20.00	190.00	0.3965	8.4081	141.9287
9	18.10	193.00	0.3320	6.1216	109.8443
10	25.30	228.00	0.1987	6.9982	102.2870
11	25.60	210.20	0.2442	6.6845	137.336
12	25.90	223.50	0.1905	6.7075	107.6633
13	27.40	227.20	0.2064	6.9328	128.2977
14	25.80	300.00	0.2143	6.7428	120.1793
15	26.70	205.50	0.2506	6.8064	130.4373
16	20.90	209.30	0.2261	8.9292	132.7520

The Results of all the three test, and the corresponding five mechanical properties are shown in Table 3.10. A comprehensive list of all the main and interaction factors for each mechanical property, is in Table 3.11. Factors concerning Damping ration are to be interpreted in the opposite way since a good laminate or a good response is the one that has less damping ration (i.e., if factor 'I' is having a positive effect as a main factor at '+' level, it means that it increases the damping at that level, whereas our objective is to reduce it).

Table 3.11 - Significant main and interaction factors for various mechanical properties.

Mechanical Properties	**Main factors**	**Interaction factors**
Shear strength	**I,A,D.**	**DH, BI, IJ, FG.**
Flexural Strength	**A,C,H**	**FI, BI, EI, GH.**
Flexural storage modulus	**B,F,I,G**	**DI, EJ, DH,HI.**
Damping (Tan delta)	**I,B,J**	**AF, FJ,AJ.**
Glass Transition Temp.	**D,B,C**	**AH, FJ, BI, CJ.**

3.4 Discussion of results.

The fact that various factors affect the mechanical properties differently is evident by looking at the summary of the factors effect in Table 3.11. This is in tune with the earlier studies [11]. There are also a quite significant number of main and interaction factors that have shown their presence in majority of the mechanical properties (Table 3.11). Main factors like A, B, C, D, I, and among interaction factors, individual factors like A, B, D, G, H, I, F, and J show influence in majority of the mechanical properties. Looking at the number of the interaction factors, it is clear that interaction effects should not be neglected while sieving out the non significant factors. In some of the experiments conducted by earlier researchers [13], the increase in the number of layers (factor H), has decreased the strength of the laminate. In this present study such an influence is not seen. Factor 'H', in the case of flexural strength has proved to increase the flexural strength at its '+' level, i.e., more the number of layers, greater the strength (Table 3.13). The increase in the strength observed in this study can be attributed to the presence of vacuum, which has proved to significantly increase the wetting of the fiber [8,9] and provide a strong bond giving rise to increased strength. In the case of shear strength, factor 'H' in interaction with 'D', has a positive influence at '-' level, but the difference in the values of shear strength when factor 'H', is at '-' level and when it is at '+' level is considerably less (Table 3.12). In the case of flexural strength, factor 'H', in interaction with factor 'G', increases the strength at its '+' level (Table 3.13). The positive effect of factor 'H' at its '+' level, is also evident in the property of flexural modulus, where higher modulus values are observed when factor 'H' is at '+' level (Table 3.14). Factor 'H' in interaction with 'A', also increases the glass transition temperature at its '+' level.

Factor 'I', as a main factor and as an individual interaction factor, at its '+' level has a very high negative influence on the response thus reducing the response (mechanical properties). This negative influence is seen in almost all of the mechanical properties (Tables 3.12, 3.13, 3.14, 3.15, and 3.16). During experimentation visual inspection has also shown the flow of chopped fibers towards the suction end. This might be due to the high resin injection pressure or due to suction at the outlet, which made the laminate fiber rich at the suction end, and resin rich at injection end. The reason for this swaying of the fibers might also be due to the less compaction of the chopped stranded matt layers, with respect to the continuous stranded matt. In order to maintain constant fiber volume fraction with respect to continuous stranded matt, which was less denser, the chopped fiber matt ended up having less preform thickness (uncompressed), hence less force exerted from top mold plate to keep the layers intact during resin injection. Looking at the analysis, factor 'I' is excluded from further examination, and is placed at '-' level, while investigating other factor. In the case of glass transition temperature factor 'I' at its '+' level is increasing the glass temperature. It is due to fact that with the swaying of the fibers towards one side of the laminate, majority part of the laminate has become resin rich. This would have aided in a perfect cure of the resin. Since glass transition temperature is a resin dependent property it would have thus increased the values of glass transition temperature.

Next factor which is of most interest is factor 'C' (pre-molding temperature) which has a positive influence on the response at its '+' level, i.e., with an increase in the pre-molding temperature there is an increase in the mechanical properties studied. This effect of factor 'C' was seen in most of the mechanical properties (Tables 3.13, 3.16). In the case of factor interaction effects where factor 'C' is involved, at '+' level it appears to have a positive influence on the response. For example, looking at interaction factor 'CJ', in the case of glass transition temperature, 'CJ' at its '+' level, increases the glass transition temperature. This interaction factor has a positive influence on the response when both the factors are either at '+' level or when both are at '-' level. The Table 3.16 shows that factor 'CJ' has a maximum average response when both the factors are at '+' level. So factor 'C' as an interaction factor also has a positive effect at its '+' level. The increase in flexural strength with an increase in pre-mold temperature, might be due to the changes in the viscosity of the resin. At high temperatures the resin will have a low viscosity thus penetrating deeper into the fiber. This would lead to a good wetting of the fibers and thus an improvement in the mechanical properties.

Summarizing the affect of factor 'C', as a main factor at its '+' level it has a positive influence on majority of the properties. Since as a main factor it has positive affect at '+' level the range for the next phase of the experiment is determined by an increase in the upper level, to a higher temperature (50 Deg.C), and the lower level is shifted to 35 Deg.C.

Table 3.12 - Analysis of main effects & Interaction effects for shear strength

Factor	Sieve 0	Effect	T -dist effect	Sieve 1	Effect	T -dist effect	Sieve 2	Effect	T -dist effect	Sieve 3	Effect	T -dist effect
I	X	-4.7953	-2.8627									
A				X	-1.9885	-1.8571						
D				X	2.7703	2.5873						
BI	X	3.2231	2.0741									
DH				X	-4.3953	-5.1523						
IJ							X	-2.5929	-2.4427			
FG							X	-1.4411	-1.3576			

Considering the main factors, factor 'A' has got a negative effect on the response at its '+' level, i.e., high injection pressure is reducing the shear strength. Factors 'I' also has a negative effect on the response at its '+' level. Only factor 'D' is having a positive effect on the response at its '+' level.

In the case of interaction factors, factor 'BI', has a positive influence on the response at its '+' level. Interaction factors 'BI', 'DH', and 'FG' have a negative influence on the response at their '+' level. An in-depth analysis of the interaction factors is shown in the following tables. The levels at which the individual factors in the interaction have a positive influence, are ascertained. In the case of interaction factor 'BI', average maximum response is noted when the individual factors are at their '+' and '-' levels respectively. So for a good response these two factors are to be kept the above said levels. In interaction factor 'DH', a good response is observed when both the factors are placed at '+' and '-' level respectively. Individual factors in interaction 'IJ' are to be placed at their '-', and '+' levels respectively, for a good response. In the case of interaction factor 'FG', a good response is observed when the individual factors are at '-' and '+' levels respectively.

Factor ‘DH’	‘-’ ‘+’	‘+’ ‘-’	‘-’ ‘-’	‘+’ ‘+’
Response	25.90	26.10	24.10	20.00
Response	25.30	25.60	23.60	27.40
Response	25.90	26.70	24.80	20.90
Response	25.80		16.00	
Response			18.70	
Response			18.10	
Average	25.72	26.13	20.88	22.77

Factor ‘BI’	‘-’ ‘+’	‘+’ ‘-’	‘-’ ‘-’	‘+’ ‘+’
Response	26.10	25.90	24.10	16.00
Response	20.00	25.80	23.60	18.10
Response		26.70	24.80	20.90
Response			18.70	
Response			25.90	
Response			25.60	
Response			27.40	
Response			25.30	
Average	23.05	26.13	24.43	18.33

Factor ‘IJ’	‘-’ ‘+’	‘+’ ‘-’	‘-’ ‘-’	‘+’ ‘+’
Response	24.1	26.1	18.7	16
Response	23.6	20	25.9	20.9
Response	24.8	18.1	25.3	
Response	25.9		25.6	
Response	27.4		26.7	
Response	25.8			
Response				
Response				
Average	25.27	21.4	24.44	18.45

Factor ‘FG’	‘-’ ‘+’	‘+’ ‘-’	‘-’ ‘-’	‘+’ ‘+’
Response	23.6	24.1	24.8	20.9
Response	18.1	18.7	26.1	26.7
Response	25.6	25.9	16	
Response	27.4		20	
Response			25.3	
Response			25.9	
Response			25.8	
Response				
Response				
Average	23.68	22.9	23.41	23.8

Table 3.13 - Analysis of main effects & Interaction effects for flexural strength

Factor	Sieve 0	Effect	T -dist effect	Sieve 1	Effect	T -dist effect	Sieve 2	Effect	T -dist effect	Sieve 3	Effect	T -dist effect
C	X	33.1140	2.4676									
A	X	-21.3195	-1.5887									
H				X	20.0459	1.5581						
FI	X	31.7933	2.6124									
BI	X	28.2017	2.3173									
EI				X	-13.9050	-1.6903						
GH				X	-22.6286	-2.7507						

In the mechanical property of flexural strength, factor 'C' at its '+' level has a positive effect on the response. Whereas 'A' at its '+' level is having a negative influence. Factor 'H' at its '+' level is also having a positive effect on the response.

In the interaction factors, factors 'FI', and 'BI', have a positive influence on the response at their '+' levels. Interaction factors, 'GH' and 'EI', have a negative influence on the response at their '+' levels. Interaction factor 'FI', tend to produce a good response if both the factors are placed at their '-' level. The individual factors in Interaction factor 'BI', are to be kept at '-' level for a good response. In Interaction factor 'GH', the individual factors are to be placed at '-' and '+' level respectively. In The case of interaction factor 'EI', the individual factors are to be placed at '+' and '-' level respectively.

Factor 'FI'	'-' '+'	'+' '-'	'-' '-'	'+' '+'
Response	176.9	220	206	209.3
Response	179	143	209	
Response	190	214.5	228	
Response	193	205.5	210.2	
Response			223.5	
Response			227.2	
Response			300	
Response				
Response				
Average	184.73	195.75	229.13	209.3

Factor 'BI'	'-' '+'	'+' '-'	'-' '-'	'+' '+'
Response	176.90	206.00	220.00	209.30
Response	179.00	143.00	209.00	
Response	190.00	228.00	214.50	
Response	193.00	210.20	227.20	
Response		223.50	300.00	
Response			205.00	
Average	184.73	202.14	229.28	209.3

Factor ‘GH’	‘-’ ‘+’	‘+’ ‘-’	‘-’ ‘-’	‘+’ ‘+’
Response	214.50	206.00	220.00	227.20
Response	190.00	193.00	209.00	209.30
Response	228.00	210.20	176.90	
Response	223.50	205.50	179.00	
Response	300.00		143.00	
Average	231.2	203.68	185.58	218.25

Factor ‘EI’	‘-’ ‘+’	‘+’ ‘-’	‘-’ ‘-’	‘+’ ‘+’
Response	176.9	223.5	220	179
Response	190	300	206	193
Response		205.5	209	209.3
Response			143	
Response			214.5	
Response			228	
Response			210.2	
Response			227.2	
Average	183.45	243	207.24	193.77

Table 3.14 - Analysis of Main effects & Interaction effects for flexural storage modulus

Factor	Sieve 0	Effect	T -dist effect	Sieve 1	Effect	T -dist effect	Sieve 2	Effect	T -dist effect	Sieve 3	Effect	T -dist effect
B	X	0.7186	3.9540									
F	X	1.0094	5.554									
I				X	0.8849	2.4466						
G				X	-0.6711	-1.8554						
DI	X	0.5121	2.2323									
EJ	X	0.5803	2.5299									
DH				X	0.4918	1.7717						
HI				X	0.4264	1.5361						

Main factors ‘B’, ‘F’, ‘G’, and ‘I’ are having an influence on the response. All the main factors except factor ‘G’, have a positive effect at their ‘+’ levels, thus improving the response. Factor ‘G’ is having a negative effect on the response at its ‘+’ level. The interaction factors that have shown some influence on the response are ‘DI’, ‘EJ’, ‘DH’, and ‘HI’. All the interaction factors have a positive influence on the response thus increasing the response. The individual factors in the interaction factor ‘DI’, ‘DH’, and ‘HI’, are to be placed at ‘+’ level for a better response, and individual factors of interaction factor ‘EJ’, are to be placed at their ‘-’ level for a better response.

Factor 'DI'	'-' '+'	'+' '-'	'-' '-'	'+' '+'
Response	7.0880	6.6845	6.9854	7.7316
Response	6.1216	6.9328	6.7694	8.4081
Response		6.8064	6.6992	8.9292
Response			8.7336	
Response			7.4504	
Response			6.9982	
Response			6.7075	
Response			6.7428	
Response				
Average	6.6048	6.8079	7.1358	8.3563

Factor 'EJ'	'-' '+'	'+' '-'	'-' '-'	'+' '+'
Response	6.9854	6.1216	7.7316	7.0880
Response	6.7694	6.8064	8.7336	6.7075
Response	6.6992		7.4504	6.7428
Response	6.9328		8.4081	8.9292
Response			6.9982	
Response			6.6845	
Average	6.8467	6.464	7.6677	7.3669

Factor 'HI'	'-' '+'	'+' '-'	'-' '-'	'+' '+'
Response	7.7316	7.4504	6.9854	8.4081
Response	7.0880	6.9982	6.7694	8.9292
Response	6.1216	6.7075	6.6992	
Response		6.9328	8.7336	
Response		6.7428	6.6845	
Response			6.8064	
Average	6.9804	6.9663	7.1131	8.6686

Factor 'DH'	'-' '+'	'+' '-'	'-' '-'	'+' '+'
Response	7.4504	7.7316	6.9854	8.4081
Response	6.9982	6.6845	6.7694	6.9328
Response	6.7075	6.8064	6.6992	8.9292
Response	6.7428		7.0880	
Response			8.7336	
Response			6.1216	
Average	6.9747	7.0742	7.0662	8.09

Table 3.15 - Analysis of main effects & Interaction effects for damping ratio

Factor	Sieve 0	Effect	T -dist effect	Sieve 1	Effect	T -dist effect	Sieve 2	Effect	T -dist effect	Sieve 3	Effect	T -dist effect
I	X	0.0479	2.0597									
B	X	-0.0429	-1.8452									
J				X	-0.0342	-1.6427						
AF	X	-0.0939	-5.5871									
FJ	X	0.0679	4.0370									
AJ				X	-0.0485	-6.4193						

The value of damping ratio, in contrast to other type of tests, has to be smaller for a good laminate. Small value of tan delta means that the sample is cured well and it is resilient. Factor 'I' has a positive influence on the response at its '+' level, hence increases the response values. Factors 'B', and 'J', are the ones that are promoting a well cured laminate. Factor 'B' has high significance at '+' level followed by 'J'.

Analyzing the interaction factors, Interaction factors 'AF', and 'AJ, at their '+' level are decreasing the damping factor. In the case factor 'FJ', it increases the response. In interaction factor 'AF', both the individual factors are to placed at their '+' level for a good response. In the case of interaction factor 'FJ', positive response is observed when the factors are placed at '+' and '-' levels. in interaction factor 'AJ', the individual factors are to place at '-' and '+' levels respectively.

Factor 'AF'	'-' '+'	'+' '-'	'-' '-'	'+' '+'
Response	0.2506	0.3965	0.2024	0.2225
Response		0.3320	0.2239	0.1680
Response			0.2312	0.2090
Response			0.2063	0.2261
Response			0.1987	
Response			0.2442	
Response			0.1905	
Response			0.2064	
Response			0.2143	
Average	0.2506	0.3643	0.2131	0.2064

Factor 'FJ'	'-' '+'	'+' '-'	'-' '-'	'+' '+'
Response	.2024	.168	.2312	.2225
Response	.2239	.209	.3965	.2261
Response	.2063	.2506	.332	
Response	.1905		.1987	
Response	.2064		.2442	
Response	.2143			
Average	0.2073	0.2092	0.2805	0.2243

. Factor 'AJ'	'-' '+'	'+' '-'	'-' '-'	'+' '+'
Response	.2024	.168	.2312	.2225
Response	.2239	.209	.1987	.2261
Response	.2063	.3965	.2442	
Response	.1905	.332	.2506	
Response	.2064			
Response	.2143			
Average	0.2073	0.2764	0.2312	0.2243

Regarding factor 'B' (Hydrocheck pressure), its presence is seen either as a main factor, or as an interaction factor, in almost all the mechanical properties (Tables 3.12, 3.13, 3.14, 3.15, and 3.16). In mechanical properties of flexural storage modulus and damping ratio, factor 'B', as a main factor, at its '+' level has a positive influence on the response. In the case of glass transition temperature it is having a negative influence on the response at its '+' level. So for a positive influence it should be placed at a '-' level or at an intermediate point in between the tested range. The interaction factor 'BI' is present in almost all the mechanical properties under observation. In interaction with 'I' (shear strength) it has shown to have a positive influence on response at its '+' level (Table 3.12). In the case of flexural strength it has a positive influence on the response at its '-' level. In mechanical property of glass transition temperature, factor 'B' has a positive influence at its '+' level. Since the range of factor 'B' has different influences on different properties, at different levels, an intermediate point between the range is necessary at which the response is maximum. This brings into notice the ideal level suggested by the manufacturer of the RTM machine. It is between 65 - 70 psi. In the second phase of the experiment factor 'B' is decided to be placed at 70 psi. Initially it was decided to include it as a factor for re-examination in the second phase. Since the injection pressure was also considered as a factor for re-examination, by reducing the hydrocheck pressure (factor 'B') to a new range the injection of resin was posing problem. Sufficient pressure was not present to inject the resin (we have already reduced the injection pressure to lower levels) into the mold. So taking all these considerations into mind factor 'B' was kept at a level decided as optimum by the RTM machine manufacturer.

Factor 'D' (post curing temperature), as a main factor, has shown considerable amount of significance in the property of glass transition temperature (Table 3.16). In the case of shear strength, at its '+' level, it increases shear strength. As an interaction factor also its presence is seen in majority of the properties. In its interaction with 'H' (shear strength, flexural storage modulus), and 'I' (flexural modulus), it has a positive influence at its '+' level, thus increasing the response. The sieve experiment has proved that if glass transition temperature is the desired property from the laminate then both the pre-mold and post curing temperatures have a significant influence on the response (the t-distribution significance of factor 'D', in the case of glass transition is around 6.4 (Table 3.16), which is highest of all the other factor significance in all the mechanical properties studied). One important concern is the range of post curing temperature the manufacturer of resin has proposed. The proposed range is in between 85 and 95 Deg.C. For improved mechanical properties, adherence to this range, and also finding an intermediate point at which there is a considerable increase in mechanical properties, is very much necessary. Large deviation from the manufacturers proposed range might cause a change in the resin chemical structure, resulting in decreased mechanical properties. Hence factor 'D' is considered for re-examination in the second phase of the experiment with the manufacturers proposed range. By performing the statistical design analysis an intermediate point may be ascertained from the range.

Injection pressure, factor 'A', as a main factor and as an interaction factor has significant effect in two mechanical properties, shear strength and flexural strength. Factor 'A' as a main factor, In both the properties, decreases the strength at its '+' level (Tables 3.12, and 3.13). One reason that could be attributed to the reduction in strength is the improper wetting of the fiber. If higher pressure of 90 psi is used along with a high level of vacuum of 10 in Hg, the pressure difference inside the mold is still around 87.5 psi. Because of this higher pressure, the flow of resin inside the mold is fast. This would cause the fast filling of the mold, even before the resin could penetrate deeply into the layers, thus ending with improper wetting of fibers. In its interaction with 'F' (damping ratio), factor 'A', has a positive effect on the response at its '+' level, whereas in interaction with 'J', it has a positive influence at its '-' level. In the case of damping ratio though it reduces the damping at '+' level, but there is less difference in the average damping ratio, when factor 'A' is at its '-' level. Since factor 'A', in majority of the mechanical properties, has a positive influence on the response at its '-' level, a further re-examination of Factor 'A' at a lower range is necessary. This would help to ascertain its influence on the mechanical properties. The range selected for the second phase is 65 - 75 psi.

Vacuum level, factor 'G', as a main factor is significant only in the case of flexural storage modulus. Here at its '+' level it decreases the modulus (Table 3.14). But as an interaction factor it has positive influence at its '+' level. In interaction with 'G', at '+' level, it has a positive effect on the shear strength. In the case of interaction with 'H' (flexural strength), it has a positive influence at its '-' level. In the visual inspection of the laminates, high concentration of bubbles were observed towards the suction end. This is due to insufficient vacuum presence to remove the voids. In the second phase of the experiment vacuum is not accommodated as a factor for re-examination, but it is held at the maximum level (25 in Hg) to minimize the void content.

Resin to catalyst ratio, factor 'J', at its '+' level increases the response, and the influence as a main factor is seen only in the case of damping ratio (Table 3.15). At this level of factor 'J' the amount of catalyst added to the resin is more, which would have aided in increase of cross linking and eventually a good cure. According to the resin manufacturer (Dow chemicals), ratios beyond 100:2 (resin to catalyst) are not advisable, since they might have a negative effect on the mechanical properties. In the interaction effects also factor 'J', in majority of the mechanical properties, increases the response at its '+' level. In its interaction with 'I', it tends to increase the shear strength. This is also evident in its interaction with 'A', and 'F', where it is reducing the damping ratio at its '+' level. Hence 'J' is kept at its '+' level while investigating other factors.

Table 3.16 - Analysis of Main effects & Interaction effects for Glass transition temp.

Factor	Sieve 0	Effect	T -dist effect	Sieve 1	Effect	T -dist effect	Sieve 2	Effect	T -dist effect	Sieve 3	Effect	T -dist effect
D	X	28.1875	6.3702									
C				X	6.5964	1.4823						
B				X	-9.6346	-2.1651						
AH	X	16.0547	2.4681									
FJ	X	12.2214	1.8788									
BI				X	13.0007	1.9960						
CJ				X	11.3799	1.7471						

Main factors 'C' and 'D' at '+' level have a positive influence on the response, while factor 'B' is having a negative effect at its '+' level. Factor 'D' is the most significant factor with a high positive influence.

Looking at the interaction factors ‘AH’, 'FJ', ‘BI’, and ‘CJ’ have got a positive influence on the response. Analyzing the interaction factors, factor ‘BI’, will produce a good response if both the individual factors are placed at their ‘+’ level. In interaction factor ‘FJ’ a good response will be possible if both factors are placed at their ‘-‘ level.

Factor ‘BI’	‘-’ ‘+’	‘+’ ‘-’	‘-’ ‘-’	‘+’ ‘+’
Response	140.4787	94.2366	109.218	132.752
Response	91.1392	83.2516	114.1487	
Response	141.9287	102.2870	124.7857	
Response	109.8443	137.336	128.2977	
Response		107.6633	120.1793	
Response			130.4373	
Average	120.8477	104.9549	121.1778	132.752

Factor ‘FJ’	‘-’ ‘+’	‘+’ ‘-’	‘-’ ‘-’	‘+’ ‘+’
Response	94.237	83.252	140.48	109.22
Response	114.15	124.79	141.93	132.75
Response	91.139	130.44	109.84	
Response	107.66		102.29	
Response	128.30		137.34	
Response	120.18			
Average	109.278	112.827	126.38	120.99

Factor 'AH'	'-' '+'	'+' '-'	'-' '-'	'+' '+'
Response	102.287	109.218	94.2366	124.7857
Response	107.6633	83.2516	114.1487	141.9287
Response	128.2977	109.8443	140.4787	132.752
Response	120.1793		91.1392	
Response			137.336	
Response			130.4373	
Response				
Response				
Average	114.6068	100.7713	117.9628	133.1555

Factor 'CJ'	'-' '+'	'+' '-'	'-' '-'	'+' '+'
Response	109.218	124.7857	140.4787	114.1487
Response	94.2366	102.287	83.2516	128.2977
Response	91.1392		141.9287	120.1793
Response	107.6633		109.8443	132.752
Response			137.336	
Response			130.4373	
Response				
Average	100.5643	113.5364	123.8794	123.8444

Stroke length, factor 'F', has shown major significance as a main factor only in the case of flexural storage modulus (Table 3.14). In the case of the flexural modulus, factor 'F', increases the response at its '+' level. The t-significance of factor 'F' in this case is very high, thus signifying its importance. As an interaction factor with 'G' (shear strength), it is again increasing the response at its '+' level. Even in the case of flexural strength it has a positive influence at its '+' level, except that the negative influence of factor 'I', at its '+' level is influencing the response (Table 3.13). In interaction with 'J', in the property of damping it reduces the damping ratio at its '+' level. The reason behind is that, in a longer stroke length ('+' level), more resin is injected into the mold in one shot. At '-' level, more short strokes are needed to fill the same volume of mold. This would result in bubbles and the formation of voids. These voids are present not at the resin front (where they can be removed by the vacuum), but towards the injection side of the laminate. Void content to a certain level may be reduced by allowing factor 'F' to stay at the '+' level while investigating other factors, and also nine (9) inch ('+' level) was the maximum pump stroke possible.

Factor 'E', valve open during the cure, is also dropped as a factor from further consideration. This is due to the lack of significant influence of this factor on the mechanical properties. This factor is significant only as an interaction factor, and at its '+' level it has a positive influence on the response (Table 3.13, and 3.14). Thus by allowing the valve to be open during the post curing, the extra resin flows out. Hence the laminate will be fiber rich, and will have improved mechanical properties. Since this is a more subjective factor, without any provision for further change in ranges, it is not considered for further examination. During the second phase of the experiment this factor is retained at its '+' level.

3.5 Conclusion

Summarizing the analysis, factors 'B', 'E','F','G','H','I', and 'J' are not chosen for further analysis and they are kept at the levels at which they showed a positive influence on the response. Factors 'A','C,'D' are considered for further analysis.

CHAPTER 4

Statistical Experimental Design Analysis.

4.1 Introduction.

The statistical design of experiment (DOE) is the process of planning the experiment so that appropriate data will be collected, which are suitable for the further statistical analysis resulting in valid and objective conclusions [12]. The application of the modern technique of design of experiments (DOE) originates from Fisher [27]. One widely used Japanese quality-improvement method, Taguchi method, has statistical design of experiments as its core. DOE is used to determine process conditions for achieving the target value and to identify factors that can be controlled to reduce variation. In this kind of setting all factors included in the experiment are varied simultaneously. The influence of unknown or non-included factors is minimized by properly randomizing the experiment. Mathematical methods are then used for the evaluation and analysis of experimental data. The mathematical model selection in DOE requires the quantitative formulation of the objective(s). Such an objective is named *the parameter of optimization* (POO), which is the result of the process under study, its output or response [12].

Each factor included in the experiment has a certain global range of variation. The basic level of the factors is termed as the *zero level (zero point),* *and the interval of factor variation* is the number which (when added to the zero level) gives the upper limit and (when subtracted from the zero level) gives the lower limit. The value of this interval is chosen as the unit of a new scale of a factor. To simplify the notation of the experiment conditions and procedure of the experimental data analysis, this new scale is chosen so that the upper limit corresponds to +*l*, lower to -*l*, and the basic level to 0. For the factors with a continuous domain, the simple transformation formula is used:

$$X_i = \frac{\bar{X}_i - X_o}{\Delta X_i} \tag{4.1}$$

Where X_i is the transformed variable.

$\bar{X}_i$ is the true value of factor.

ΔX_i is the interval of the factor variation (in true units).

X_0 is the zero level of the factor.

The minimum number of factors levels used in this study is two. The two factor values corresponding to the upper and lower limits of the interval of variation are used as the two levels. These values are called the upper and lower levels and are designated "+*l*' and "-*l*' (or even simpler, "+" and "-"). When two factor levels are used in DOE, the design plan is designated as 2^k, where k is the number of involved factors [28].

4.2 2^k factorial experiment, complete block

Experiments are conducted in order to investigate the effects of one or more factors on a response. When an experiment involves two or more factors, the factors can influence the response individually or jointly. Often, as in the case of one factor-at-a time experimentation, an experimental design does not allow one to properly assess the joint effects of the factors.

Factorial experiments conducted in completely randomized designs are especially useful for evaluating joint factor effects. Factorial experiments include all possible factor level combinations in the experimental design. Completely randomized designs are appropriate when there are no restrictions on the order of the testing or when all the experimental units to be used in the experiment can be regarded as homogeneous.

The mathematical description of the object under study in the vicinity of the zero point can be obtained by varying each factor at two levels, distinguished from the zero level by the interval of variation. When the experiment includes all possible non-repeated factor-level combinations, such experiment is termed as *the complete block.* The number of such combinations is $N = 2^k$.

When using the 2^3 factorial experiment (complete block), the regression equation is:

$$\hat{y} = \hat{E} = b_0 + \sum_{i=1}^{3} b_i \bar{X}_i + \sum_{i,j} b_{ij} \bar{X}_i \bar{X}_j + b_{123} \bar{X}_1 \bar{X}_2 \bar{X}_3 \qquad (4.2)$$

where, $(i \neq j)$

The complete block enables an experimentalist to obtain the separate, statistically independent estimations for all coefficients in Eq. (4.2). This is the main advantage of such a type of experiment.

Obtaining the mathematical model in form of Eq (4.2) includes several successive stages:

(1) design of experiment: statement of a problem; choice of POO; selection of factors to be involved; choice of the levels of these factors; selection of the sequence of the factor-level combinations; selection of the number of observations to be taken; selection of the order of test to be used; selection of the method of randomization to be used; selection of a mathematical model to describe the experimental results.

(2) experiment itself has a series of tests; data collection in each test; evaluation and analysis; examination of the statistical significance of the model coefficients; examination of homogeneity of the row variances; examination of the adequateness of the obtained mathematical model. An important step at stage (1) is the selection of the order of test to be used.

4.3 Design matrix

Using the code values (+*l*, -*l*), the experiment conditions can be written as a table, called *the design matrix,* where the rows correspond to different tests and the columns correspond to different code values of the factors [12, 27 -33].

In the case of three included factors, a design matrix is as shown in Table 4.1. In this table, columns X_1, X_2, X_3 form the design matrix because they directly set up the tests conditions. Further, the columns for the interactions: X_1X_2, X_1X_3, X_2X_3, $X_1X_2X_3$, are placed in Table 4.1, and they are used for the estimation of the factors interactions. An artificial variable X_0 is added to the table and used to estimate the coefficient b_o. Tables contain such a column are called the *extended design matrices.*

Table 4.1-Treatment combinations and effects in 2^3 factorial experiment.

Point of the matrix	X_0	X_1	X_2	X_3	X_1X_2	X_1X_3	X_2X_3	$X_1X_2X_3$
1	+	-	-	-	+	+	+	-
2	+	+	-	-	-	-	+	+
3	+	-	+	-	-	+	-	+
4	+	+	+	-	+	-	-	-
5	+	-	-	+	+	-	-	+
6	+	+	-	+	-	+	-	-
7	+	-	+	+	-	-	+	-
8	+	+	+	+	+	+	+	+

The following notation of tests is used. A number u (u = *1-8) is attributed to each point in the design matrix.* The tests have double numbering: the first number shows the point in the design matrix; the second is the test number at this point. The number of parallel tests at the same point is denoted as r_U (r_U >1). For example, y_{uj}; is the response, obtained in *j-th* test conducted at u-th point.

4.4 Experimental study using DOE.

DOE is considered for the experimental study of the influence of three parameters, namely, Injection pressure, A(X_1), pre-molding temperature, C(X_2), and curing temperature, D(X_3), on shear strength, flexural strength, flexural storage modulus, damping ratio, and glass transition temperature. Thus, the factorial experiment 2^3 complete block was used. Referring to Eq. (4.2), the mathematical model for this case can be written in transformed variables as follows:

$$\hat{y} = b_0+b_1X_1+b_2X_2+b_3X_3+b_{12}X_1X_2+b_{13}X_1X_3+b_{23}X_2X_3+b_{123}X_1X_2X_3. \qquad (4.2)$$

The levels of factors and the intervals of factors variation are shown in Table 4.2.

4.5 POO measurements and recording.

At each point of factorial space the experiments were conducted genuinely four times ($r = 4$). The sequence of tests was arranged in accordance with a table of random numbers. Experimental results for shear strength are shown in Tables 4.3.

Table 4.2 - levels of factors and their intervals of variation (for all tests).

Levels of factors.	Notation	A, psi X_1	C, °C X_2	D, °C X_3
Basic	**0**	**72.5**	**42.5**	**87.5**
Interval of variation	ΔX_i	**2.5**	**7.5**	**7.5**
Upper	***+I***	**75**	**50**	**95**
Lower	***-I***	**70**	**35**	**80**

Table 4.3 - Test results (Shear strength, MPa).

Points of matrix, u	y_{u1}	y_{u2}	y_{u3}	y_{u4}	y_u Average response	s^2_u Row Variance
1	25.75534	26.87968	26.52146	26.64082	26.44933	0.23623
2	30.07251	28.84618	27.43442	28.59474	28.73696	1.17082
3	26.70500	32.47600	30.19121	30.77867	30.03772	5.87493
4	33.47181	27.23586	30.46973	28.68389	29.96532	7.21397
5	31.03318	32.02890	29.19618	29.38875	30.41175	1.84183
6	32.39764	31.85600	28.89839	30.16004	30.82802	2.56337
7	28.14154	25.93042	27.56586	29.47137	27.77730	2.15274
8	31.83774	31.59750	29.84311	31.46475	31.18577	0.82505
$s^2\{y\}= \sum s^2_u/8 = 2.73487$						

4.6 *Obtaining the mathematical model.*

The objective of the experiment is to obtain the mathematical model which reflects the phenomenon under study. The detailed procedure for the mathematical model obtained here is presented only for shear strength data, Table 4.3. For all the other tests only the results are shown.

The orthogonality of the design matrix allow simplification of the calculations of regression coefficients and is a distinguished advantage of the considered methods of experimental design. It is known [27] that for any number of factors, the regression coefficients can be calculated by using the following formula:

$$b_i = \frac{\sum_{u=1}^{n} X_{iu} \bar{y}_u}{n} \tag{4.3}$$

Here, 'i' is the index of the coefficient, where i=0,1,2,3,12,23,13,123. X_{iu} is the transformed variable determined by Table 4.1. k is the number of the factor; $\bar{y}_u$ is the average response at the considered point, and is calculated using:

$$\bar{y}_u = \frac{\sum_{j=1}^{r} \bar{y}_{uj}}{r} \tag{4.4}$$

Here 'r' is the number of the repeated tests at one point in the matrix. Since each factor (except X_0) is varied at two levels (+*l*, and -*l*), the calculations are conducted by attributing to the entries of column y_u the signs of the entries of column for corresponding factor, followed by algebraic summation of these entries. A regression coefficient is obtained by dividing the result by the number of plan points. In the examined case Table 4.3:

$$b_1 = \frac{\sum_{u=1}^{8} X_{1u}\bar{y}_u}{8}$$

b_1 = 1/8(- 26.44933 + 28.73696 - 30.03772 + 29.96532 - 30.41175 + 30.82802 - 27.77730 + 31.18577) = 0.754998.

Similarly b_2 is calculated as,

$$b_2 = \frac{\sum_{u=1}^{8} X_{2u}\bar{y}_u}{8}$$

b_2 = 1/8(- 26.44933 - 28.73696 + 30.03772 + 29.96532 - 30.41175 - 30.82802 + 27.77730 + 31.18577) = 0.317507.

$$b_3 = \frac{\sum_{u=1}^{8} X_{3u}\bar{y}_u}{8}$$

b_3 = 1/8(- 26.44933 - 28.73696 - 30.03772 - 29.96532 + 30.41175 + 30.82802 + 27.77730 + 31.18577) = 0.626689.

$$b_{12} = \frac{\sum_{u=1}^{8} X_{12u}\bar{y}_u}{8}$$

b_{12} = 1/8(+ 26.44933 - 28.73696 - 30.03772 + 29.96532 + 30.41175 - 30.82802 - 27.77730 + 31.18577) = 0.0790224.

$$b_{13} = \frac{\sum_{u=1}^{8} X_{13u}\bar{y}_u}{8}$$

b_{13} = 1/8(+ 26.44933 - 28.73696 + 30.03772 - 29.96532 - 30.41175 + 30.82802 - 27.77730 + 31.18577) = 0.201188.

$$b_{23} = \frac{\sum_{u=1}^{8} X_{23u}\bar{y}_u}{8}$$

b_{23} = 1/8(+ 26.44933 + 28.73696 - 30.03772 - 29.96532 - 30.41175 - 30.82802 + 27.77730 + 31.18577) = - 0.886682.

$$b_{123} = \frac{\sum_{u=1}^{8} X_{123u}\bar{y}_u}{8}$$

b_{123} = 1/8(- 26.44933 + 28.73696 + 30.03772 - 29.96532 + 30.41175 - 30.82802 - 27.77730 + 31.18577) = 0.669031.

Calculation of b_0 is conducted using the same rule :

$$b_0 = \frac{\sum_{u=1}^{8} X_{0u}\bar{y}_u}{8}$$

b_0 = 1/8(+ 26.44933 + 28.73696 + 30.03772 + 29.96532 + 30.41175 + 30.82802 + 27.77730 + 31.18577) = 29.424.

Then the regression equation in the transformed variables becomes :

$$\hat{y} = 29.24 + 0.754998\ X_1 + 0.317507\ X_2 + 0.626689\ X_3 + 0.0790224\ X_1X_2 + 0.201188\ X_1X_3 - 0.886682\ X_2X_3 + 0.669031\ X_1X_2X_3. \qquad (4.5)$$

4.7 Statistical examination of the results.

Since DOE originates from the statistical nature of studying process, the resulting equation has to go through a careful statistical analysis. The objective of the analysis is dual : on one hand it is necessary to extract the maximum information from the collected data, on the other hand, to check the reliability and accuracy of the obtained results. The following procedure of the experimental data examination is done for this study :

(1) Calculation of the row variances and variance of the response.

The row variances are calculated using the data from the table (4.2) by the following formula:

$$s^2_u = \frac{\sum_{j=1}^{r}(y_{uj}-\bar{y}_u)^2}{r-1} \qquad (4.6)$$

$s^2_1 = \{(25.75534-26.44933)^2 + (26.87968-26.44933)^2 + (26.52146-26.44933)^2 +$

$(26.64082-26.44933)^2\} / (4-1) = 0.236231.$

$s^2_2 = \{(30.07251-28.73696)^2 + (28.84618-28.73696)^2 + (27.43442-28.73696)^2 +$

$(28.59474-28.73696)^2\} / (4-1) = 1.17082.$

$s^2_3 = \{(26.70500-30.03772)^2 + (32.47600-30.03772)^2 + (30.19121-30.03772)^2 +$

$(30.77867-30.03772)^2\} / (4-1) = 5.87494.$

$s^2_4 = \{(33.47181-29.96532)^2 + (27.23586-29.96532)^2 + (30.46973-29.96532)^2 +$

$(28.68389-29.96532)^2\} / (4-1) = 7.21397.$

$s^2_5 = \{(31.03318-30.41175)^2 + (32.02890-30.41175)^2 + (29.19618-30.41175)^2 +$

$(30.41175-30.41175)^2\} / (4-1) = 1.84183.$

$s^2_6 = \{(32.39764-30.82802)^2 + (31.85600-30.82802)^2 + (28.29839-30.82802)^2 +$

$(30.16004-30.82802)^2\} / (4-1) = 2.56337.$

$$s^2_7 = \{(28.14154-27.77730)^2 + (25.93042-27.77730)^2 + (28.29839-27.77730)^2 + (30.16004-27.77730)^2\} / (4-1) = 2.15274.$$

$$s^2_8 = \{(31.83774-31.18577)^2 + (31.59750-31.18577)^2 + (29.84311-31.18577)^2 + (31.46475-31.18577)^2\} / (4-1) = 0.825051.$$

The results of the calculations are shown in Table 4.2.

The variance of the response $s^2\{y\}$, is the arithmetical average of n different variants of tests (that is the average variance), i.e.,

$$s^2\{y\} = \frac{\sum_{u=1}^{n}(s^2_u)}{n} \qquad (4.7)$$

For the considered case (using the data from Table 7.2) $s^2\{y\} = 2.73487$.

(2) Examination of variance homogeneity.

The examination is conducted using the statistical criteria of *Fisher, Cochran, and Barlett.* It should be pointed out that *Fisher's, F-criterion,* was usually used for the examination of variance homogeneity; and this is a common mistake in the statistical analysis of the experimental data. *F-criterion* cannot be used when the number of the examining variances are greater than two, because this criterion takes into consideration only the maximum and minimum variances but ignores the others. When numbers of test repetitions at each point of design plan are identical, Cochran criterion should be used. This criterion is calculated as a ratio of the maximum variance, s^2_{umax}, to the sum of all variances. In the considered case:

$$G = \frac{7.21397}{21.878952} = 0.329722. \tag{4.8}$$

Using the table of Cochran numbers, the critical Cochran number is found to be $G_{Cr} = 0.60$, for $f_{umax} = 2$ and $f_{den} = 32$ (the total number of the tests) degrees of freedom and at 5% level of significance. Since the experimentally defined value of G is less than G_{cr}, the variances are considered homogeneous.

(3) Regression analysis.

The mathematical model was defined using the least squares method. This method was used as a computational method. Now it is necessary to conduct the statistical estimation of the obtained mathematical model. Normally the regression analysis is based on the following principles:

- the observed results $y_1, y_2 \ldots\ldots y_n$ of response of n points of the factorial space are independent, normally distributed random values;
- the variance of response does not depend on the absolute value of y and the value of factors. In another word, variances at different points of the design matrix are homogeneous. The validity of this principle is shown above;

- the values of factors are not random. In practice it means that the errors of measurements of the independent variables X_1, X_2...... X_k are much less than the errors of factors reproduced.

(4) Examination of significance of the model coefficients.

Examination of the significance of each model coefficient is conducted independently. Thus, the examination by employing *the t-criterion (Student's* criterion) can be used. While using the complete factorial experiment, the confidence intervals for all coefficients are equal. First of all, the variance of the regression coefficient, $s^2\{b_i\}$ have to be defined. Under uniform repetition of test, with a number 'r' of repetitions at each point, this variance can be defined as:

$$s^2\{b_i\} = s^2(y)/nr. \tag{4.9}$$

with $f_E = n(r - l)$ degrees of freedom.

In the considered case:

$$s^2\{b_i\} = \frac{2.73487}{32} = 0.0855 \quad s\{b_i\} = \sqrt{0.0855} = 0.2924.$$

it can be seen from this formula that the variances of all coefficients are equal, because they depend only on the error of experiment itself and on the number of repetitions. Using these data, *t_i-criterion* can be calculated by the following formula :

$$t_i = \frac{|b_i|}{s\{b_i\}} \tag{4.10}$$

$$t_0 = \frac{29.424}{0.292343} = 100.649; \qquad t_1 = \frac{0.754998}{0.292343} = 2.58257;$$

$$t_2 = \frac{0.317507}{0.292343} = 1.08608; \qquad t_3 = \frac{0.626689}{0.292343} = 2.14367;$$

$$t_{12} = \frac{0.0790224}{0.292343} = 0.270307; \qquad t_{13} = \frac{0.201188}{0.292343} = 0.688191;$$

$$t_{23} = \frac{0.886682}{0.292343} = 3.03301; \qquad t_{123} = \frac{0.669031}{0.292343} = 2.28851;$$

The critical value of *t-criteria,* t_{cr} *is* defined with $n(r - 1) = 24$ degrees of freedom and at significant level of 5%. In the considered case $t_{cr} = 1.711$. Now. if $t_i < t_{cr}$ then a coefficient b_i is considered to be insignificant, that is $b_i=0$. In the considered case, coefficients b_2, b_{12}, b_{13} are found to be insignificant. A confidence interval for each significant coefficient has to be determined. The length of such interval is $2\Delta b_i$, where

$$\Delta b_i = t_{cr}\, s\{b_i\} = 0.5 \tag{4.11}$$

A coefficient b_i is considered to be significant if its absolute value is more than a half of the length of the corresponding confidence interval. The orthogonal matrix allows one to define the confidence interval separately for each coefficient of regression, and when any coefficient is found to be insignificant it can be rejected without revaluation of the others. Now, the mathematical model is represented by the equation including only significant coefficients. In the considered case:

$$\hat{y} = 29.24 + 0.754998\ X_1 + 0.626689\ X_3 - 0.886682\ X_2X_3 + 0.669031\ X_1X_2X_3 \qquad (4.12)$$

To obtain the model in real values of variables, it is necessary to make the reverse transformation from Eq.(4.1), where we have,

$$X_1 = (\tilde{X}_1 - 72.5)/2.5; \quad X_2 = (\tilde{X}_2 - 42.5)/7.5; \quad X_3 = (\tilde{X}_3 - 87.5)/7.5;$$

By substituting the above values into Eq (4.12) we obtain :

$$\hat{y} = -1341.08 + 17.9941\ A + 31.56\ C + 15.4127\ D - 0.416\ AC - 0.361\ CD - 0.202\ AD + 0.00475755\ ACD \qquad (4.13)$$

Equation (4.13) reveals that in the present case, and for the chosen upper and lower factor limits, the shear strength depends on injection pressure, pre-mold temperature, post curing temperature, and also on their interaction. Therefore the influence of these parameters on shear strength cannot be judged individually as it was considered before [1,2].

At this stage of the statistical analysis, the common problem is the insignificance of the particular regression coefficient(s) which were thought to be significant before testing. The statistical insignificance of a regression coefficient can be caused by the following reasons:

1. The chosen zero level X_{io} is too close to the point of particular extreme of variable X_i.
2. The chosen interval of variation ΔX_i is not large enough to detect the influence of X_i on POO;
3. Factor (or factors product) corresponding to the coefficient does not have the functional relation with POO;
4. Conducted experiment has a large error due to the presence of uncontrollable variables.

When it is believed that the considered factor has a significant influence on POO then

1. In the first case, the basic value of the factor should be changed .
2. In the second case, the interval of variation ΔX_i, should re-set to be bigger.
3. In the third case, it is necessary to re-do the experiment, trying to reduce errors.

(5) Adequateness of the model.

The next step, after calculating the model coefficients, is to check the adequateness of the obtained model. Therefore, the estimation of the deviations between *POO*, predicted by the obtained model, and the experimental value of *POO* at the same matrix points should be determined. This deviation is called the residual variance or variance of adequateness, s^2_{ad}. The estimation of s^2_{ad}, (when the number of the repeated tests at each point in the design plan is the same) can be found as:

$$s^2_{ad} = \frac{r}{n-m} \sum_{u=1}^{n} (\bar{y}_u - \hat{y}_u)^2 \qquad (4.14)$$

Here 'm' is the number of terms (Eq.(4.12)) in the obtained model including the free term; $\bar{y}_u$ is the average response at point u; $\hat{y}_u$ is the calculated (Eq. (4.12)) response at the same point. The variance of adequateness is defined with the degrees of freedom equal to:

$$f_{ad} = n - m = 8 - 5 = 3 \qquad (4.15)$$

The examination of the model adequateness includes the definition of the ratio between the variance of adequateness s^2_{ad}, and the variance of reproduction, $s^2\{y\}$. The procedure includes using the *F-criterion* of Fisher. This criterion provides the examination, called "null-hypothesis", of the equality of these two variances, and this criterion is formulated as the following ratio:

$$F = s^2_{ad} / s^2\{y\} \qquad (4.16)$$

If the calculated value *of 'F'* is less than 'F_{cr}', where 'F_{cr}' is defined using a statistical table for corresponding degrees of freedom.

$$f_{ad} = n - m \text{ and } f_E = n(r - l). \quad (4.17)$$

and certain level of significance, α%, then the model is recognized to be adequate to the process under study. Otherwise, the model is recognized to be inadequate.

If the variance of adequateness, s^2_{ad}, does not exceed the variance of reproduction, $s^2\{y\}$, then $F \leq F_{cr}$ is valid for any number degrees of freedom.

Calculation of the responses:

$\hat{y}_1$ = 29.424-0.754998-0.626689-0.886682-0.669031 = 26.4866.

$\hat{y}_2$ = 29.424+0.754998-0.626689-0.886682+0.669031= 29.3347.

$\hat{y}_3$ = 29.424-0.754998-0.626689+0.886682+0.669031 = 29.598.

$\hat{y}_4$ = 29.424+0.754998-0.626689+0.886682-0.669031 = 29.7700.

$\hat{y}_5$ = 29.424-0.754998+0.626689+0.886682+0.669031 = 30.8514.

$\hat{y}_6$ = 29.424+0.754998+0.626689+0.886682-0.669031 = 31.0234.

$\hat{y}_7$ = 29.424+0.754998+0.626689-0.886682-0.669031 = 27.7400.

$\hat{y}_8$ =29.424+0.754998+0.626689-0.886682+0.669031 = 30.5881.

The result of calculations of ($\bar{y}_u - \hat{y}_u$) is shown in Table 4.4. Now estimating

$$s^2_{ad} = \frac{r}{n-m}\sum_{u=1}^{n} (\bar{y}_u - \hat{y}_u)^2 = 1.57367 \qquad F=0.57541.$$

Table 4.4 - Calculation of the estimated response (shear strength)

Points of matrix-u	$\bar{y}_u$	$\hat{y}_u$	$(\bar{y}_u - \hat{y}_u)^2$
1	26.44933	26.4866	**0.00139**
2	28.73696	29.3347	**0.35729**
3	30.03772	29.598.	**0.19335**
4	29.96532	29.7700.	**0.03815**
5	30.41175	30.8514.	**0.19329**
6	30.82802	31.0234	**0.03817**
7	27.77730	27.7400	**0.00139**
8	31.18577	30.5881	**0.35721**

Since in the considered case $s^2_{ad} < s^2\{y\}$, then the model adequateness is obvious even without using *F-criterion* calculations.

The examination of the model adequateness is possible only when $f_{ad}>0$. If the number of matrix points is equal to the number of the estimated coefficients of the obtained model *(n = m)* then there is no degrees of freedom (f_{ad}= 0) to examine the null-hypothesis.

4.8 Experimental study of flexural strength using DOE.

The results of the experimental study of flexural strength are shown in Table 4.5.

Table 4.5 - Test results (flexural strength, MPa)

Points of matrix u	y_{u1}	y_{u2}	y_{u3}	y_{u4}	y_u Average response	s^2_u Row variance
1	237.233	251.708	250.413	265.838	251.298	136.729
2	258.59	270.589	266.549	274.816	267.636	47.7615
3	325.067	275.539	262.566	251.376	278.637	1055.59
4	300.797	312.586	273.596	293.8	295.195	267.431
5	301.418	293.658	289.976	312.843	299.474	102.182
6	273.07	266.74	273.656	285.761	274.807	63.1361
7	317.807	293.664	298.04	324.605	308.529	225.169
8	304.85	283.062	306.597	282.344	294.213	177.242
$s^2\{y\} = \Sigma s^2_u/8 = 259.405$						

Examination of row variances :

$s^2\{y\} = \Sigma s^2_u/8 = 259.405.$

G = 0.50866

G is less than G_{cr} (0.6), so the variances are considered to be homogeneous

Calculation of coefficients of regression equation:

b_0 = 283.724

b_1 = -0.760872

b_2 = 10.4199

b_3 = 10.5321

b_{12} = 1.32138

b_{13} = -8.98481

b_{23} = -3.30451

$b_{123} = 1.26643$

Examination of significance of the model coefficients :

$s^2\{b_i\} = 2.84718$, with $f_E = 24$ degrees of freedom.

$t_0 = 99.6508$ (not less than t_{cr}), so SIGNIFICANT

$t_1 = 0.267237$ (less than t_{cr}), and so INSIGNIFICANT

$t_2 = 3.65974$ (not less than t_{cr}), so SIGNIFICANT

$t_3 = 3.63915$ (not less than t_{cr}), so SIGNIFICANT

$t_{12} = 0.464102$ (less than t_{cr}), and so INSIGNIFICANT

$t_{13} = 3.15569$ (not less than t_{cr}), so SIGNIFICANT

$t_{23} = 1.16063$ (less than t_{cr}), and so INSIGNIFICANT

$t_{123} = 0.444803$ (less than t_{cr}), and so INSIGNIFICANT

The accepted regression terms before the conversion :

283.724	(TAKEN)
-0.760872	X1 (NOT TAKEN)
10.4199	X2 (TAKEN)
10.5321	X3 (TAKEN)
1.32138	X1 X2 (NOT TAKEN)
-8.98481	X1 X3 (TAKEN)
-3.30451	X2 X3 (NOT TAKEN)
1.26643	X1 X2 X3 (NOT TAKEN)

Mathematical model in true transformed variables is :

$$y = 283.724 + 10.4199\,X2 + 10.5321\,X3 - 8.98481X1X3 \tag{4.18}$$

Mathematical model in true real variables is :

$$Y = -2938.06 + 41.9291A + 1.38933C + 36.1455D - 0.47919AD \tag{4.19}$$

Examination of the model adequateness :

The estimated averages of the responses are shown in Table 4.6:

The variance of adequateness : $s^2_{ad} = 118.789 < s^2\{y\} = 259.405$.

$f_{ad} = n - m = 8 - 4 = 4$

F-Criterion : Since $F < F_{cr}$ the model is adequate.

Table 4.6 - Estimated averages of the response (flexural strength)

Points of matrix-u	$\bar{y}_u$	$\hat{y}_u$	$(\bar{y}_u - \hat{y}_u)^2$
1	**251.298**	**253.787**	**6.195**
2	**267.636**	**271.756**	**16.974**
3	**278.637**	**274.627**	**16.08**
4	**295.195**	**292.596**	**6.755**
5	**299.474**	**292.821**	**44.262**
6	**274.807**	**274.851**	**0.002**
7	**308.529**	**313.66**	**26.327**
8	**294.213**	**295.691**	**2.184**

4.9 Experimental study of Glass transition temperature using DOE.

The results of the experimental study of Glass transition temperature are shown in Table 4.7.

Examination of row variances :

$s^2\{y\} = \Sigma s^2_u/8 = 1.81288.$

G = 0.395865.

G is less than G_{cr} (0.6), so the variances are considered to be homogeneous

Table 4.7 - Test results (Glass transition temperature, °C)

Points of matrix u	y_{u1}	y_{u2}	y_{u3}	y_u Average response	s^2_u Row Variance
1	87.03	87.65	86.17	86.95	0.552402
2	82.27	85.14	85.06	84.1567	2.67124
3	91.87	90.84	90.81	91.1733	0.364237
4	102.11	100.59	100.26	100.987	0.973634
5	124.24	119.45	121.72	121.803	5.74123
6	122.16	120.18	119.45	120.597	1.96624
7	123.88	123.72	121.56	123.053	1.67894
8	115.66	117.15	116.39	116.40	0.555098
$s^2\{y\} = \Sigma s^2_u/8 = 1.81288$					

Calculation of coefficients of regression equation:

$b_0 = 105.64$

$b_1 = -0.105$

$b_2 = 2.26334$

$b_3 = 14.8233$

$b_{12} = 0.895$

$b_{13} = -1.86$

$b_{23} = -3.00$

$b_{123} = -2.25666$

Examination of significance of the model coefficients :

$s^2\{b_i\} = 0.274839$, with $f_E = 16$ degrees of freedom.

$t_0 = 384.37$ (not less than t_{cr}), so SIGNIFICANT

$t_1 = 0.382043$ (less than t_{cr}), and so INSIGNIFICANT

$t_2 = 8.23513$ (not less than t_{cr}), so SIGNIFICANT

$t_3 = 53.9346$ (not less than t_{cr}), so SIGNIFICANT

$t_{12} = 3.25645$ (not less than t_{cr}), so SIGNIFICANT

$t_{13} = 6.76761$ (not less than t_{cr}), so SIGNIFICANT

$t_{23} = 10.9155$ (not less than t_{cr}) so SIGNIFICANT

$t_{123} = 8.21085$ (not less than t_{cr}), so SIGNIFICANT

The accepted regression terms before the conversion :

105.64	(TAKEN)
-0.105	X1 (NOT TAKEN)
2.26334	X2 (TAKEN)
14.8233	X3 (TAKEN)
0.895	X1 X2 (TAKEN)
-1.86	X1 X3 (TAKEN)
-3.00	X2 X3 (TAKEN)
-2.25666	X1 X2 X3 (TAKEN)

Mathematical model in transformed variables is :

$$y = 105.64 + 2.26334X_2 + 14.8233X_3 + 0.895X_1X_2 - 1.86X_1X_3 - 3X_2X_3 - 2.25666X_1X_2X_3 \quad (4.20)$$

Mathematical model in real variables is :

$$Y = 3565.84 - 53.0249A - 100.293C - 38.0109D + 1.4517\,AC + 0.5828\,AD + 1.11\,CD - 0.01605\,ACD. \qquad (4.21)$$

Examination of the adequateness of the model :

The estimated averages of the responses are shown in Table 4.8:

The Variance of adequateness : $s^2_{ad} = 0.264602 < s^2\{y\} = 1.81288.$

$f_{ad} = n - m = 8 - 7 = 1$

F-Criterion : since $F<F_{cr}$ the model is adequate.

Table 4.8 - Estimated averages of the response (Glass transition temperature)

Points of matrix-u	$\bar{y}_u$	$\hat{y}_u$	$(\bar{y}_u - \hat{y}_u)^2$
1	86.95	86.845	**0.011**
2	84.1567	84.2617	**0.011**
3	91.1733	91.0684	**0.011**
4	100.987	101.092	**0.011**
5	121.803	121.698	**0.011**
6	120.597	120.702	**0.011**
7	123.053	122.948	**0.011**
8	116.40	116.505	**0.011**

4.10 Experimental study of flexural storage modulus using DOE.

The results of the experimental study of flexural storage modulus are shown in Table 4.9.

Examination of row variances :

$s^2\{y\} = \Sigma\, s^2_u/8 = 0.0294313.$

$G = 0.428069$.

G is less than G_{cr} (0.6), so the variances are considered to be homogeneous

Calculation of coefficients of regression equation:

$b_0 = 8.13962$

$b_1 = -0.103875$

$b_2 = 0.264958$

$b_3 = 0.114708$

$b_{12} = 0.168458$

$b_{13} = -0.213792$

$b_{23} = -0.106458$

$b_{123} = 0.0940418$

Examination of significance of the model coefficients :

$s^2\{b_i\} = 0.0350186$, with $f_E = 16$ degrees of freedom.

$t_0 = 232.437$ (not less than t_{cr}), so SIGNIFICANT

$t_1 = 2.96627$ (not less than t_{cr}), so SIGNIFICANT

$t_2 = 7.56621$ (not less than t_{cr}), so SIGNIFICANT

$t_3 = 3.27564$ (not less than t_{cr}), so SIGNIFICANT

$t_{12} = 4.81053$ (not less than t_{cr}), so SIGNIFICANT

$t_{13} = 6.10509$ (not less than t_{cr}), so SIGNIFICANT

$t_{23} = 3.04005$ (not less than t_{cr}), so SIGNIFICANT

$t_{123} = 2.68548$ (not less than t_{cr}), so SIGNIFICANT

Table 4.9 - Test results (flexural storage modulus, GPa)

Points of matrix u	y_{u1}	y_{u2}	y_{u3}	y_u Average response	s^2_u Row variance
1	7.716	7.664	7.474	7.618	0.016228
2	7.477	7.536	8.054	7.689	0.100789
3	8.157	8.306	8.173	8.212	0.0066909
4	8.715	8.502	8.525	8.58067	0.0136664
5	8.695	8.545	8.788	8.676	0.015033
6	7.47	7.599	7.478	7.51567	0.005224
7	8.497	8.535	8.372	8.468	0.0072730
8	8.633	8.337	8.103	8.35767	0.0705455
$s^2\{y\} = \sum s^2_u/8 = 0.0294313$					

The accepted regression terms before the conversion :

8.13962	(TAKEN)
-0.103875	X1 (TAKEN)
0.264958	X2 (TAKEN)
0.114708	X3 (TAKEN)
0.168458	X1 X2 (TAKEN)
-0.213792	X1 X3 (TAKEN)
-0.106458	X2 X3 (TAKEN)
0.0940418	X1 X2 X3 (TAKEN)

Mathematical model in transformed variables is :

$$y = 8.13962 - 0.103875 X_1 + 0.264958 X_2 + 0.114708 X_3 + 0.168458 X_1X_2 - 0.213792 X_1X_3 - 0.106458 X_2X_3 + 0.094081 X_1X_2X_3. \qquad (4.22)$$

Mathematical model in real variables is :

$$Y = -232.82755 + 3.0745\,A + 3.812\,C + 3.048085\,D - 0.0497707\,AC - 0.0401892\,AD - 0.050878\,CD + 0.0006755\,ACD. \qquad (4.23)$$

Examination of the adequateness of the model :

The estimated averages of the responses are shown in Table 4.10:

The Variance of adequateness : $s^2_{ad} = 0$; $s^2\{y\} = 0.0294313$.

$f_{ad} = n - m = 8 - 8 = 0$; F-Criterion : Since $F < F_{cr}$ the model is adequate.

Table 4.10 - Estimated averages of the response (flexural storage modulus)

Points of matrix-u	$\bar{y}_u$	$\hat{y}_u$	$(\bar{y}_u - \hat{y}_u)^2$
1	7.618	**7.618**	**0.0**
2	7.689	**7.689**	**0.0**
3	8.212	**8.212**	**0.0**
4	8.58067	**8.58067**	**0.0**
5	8.676	**8.676**	**0.0**
6	7.51567	**7.51567**	**0.0**
7	8.468	**8.468**	**0.0**
8	8.35767	**8.35767**	**0.0**

4.11 Experimental study of damping ratio using DOE.

The results of the experimental study of damping ratio are shown in Table 4.11.

Examination of row variances :

$s^2\{y\} = \Sigma\, s^2_u/8 = 6.17719e\text{-}05$.

$G = 0.215907$.

G is less than G_{cr} (0.6), so the variances are considered to be homogeneous.

Table 4.11 - Test results (damping ratio)

Points of matrix u	y_{u1}	y_{u2}	y_{u3}	y_{u4}	y_u Average response	s^2_u Row variance
1	0.1362	0.1424	0.1315	0.1316	0.135425	2.65e-05
2	0.1445	0.1571	0.1395	0.1513	0.1481	5.94e-05
3	0.1467	0.1481	0.1388	0.1635	0.149275	0.000107
4	0.1313	0.1358	0.141	0.1376	0.136425	1.63e-05
5	0.1461	0.1572	0.1685	0.1607	0.158125	8.66e-05
6	0.1686	0.1734	0.1815	0.1629	0.1716	6.20e-05
7	0.1607	0.1474	0.1569	0.1539	0.154725	3.16e-05
8	0.1605	0.1572	0.1739	0.1493	0.160225	0.000105
	$s^2\{y\} = \sum s^2_u/8 = 6.17719\text{e-}05$					

Calculation of coefficients of regression equation:

b_0 = 0.151737

b_1 = 0.00235

b_2 = -0.001575

b_3 = 0.00943124

b_{12} = -0.0041875

b_{13} = 0.00239375

b_{23} = -0.00211875

b_{123} = 0.00219375

Examination of significance of the model coefficients :

$s^2\{b_i\}$ = 0.00138938, with $f_E = 24$ degrees of freedom.

t_0 = 109.213 (not less than t_{cr}), so SIGNIFICANT

t_1 = 1.69141 (less than t_{cr}), and so INSIGNIFICANT

t_2 = : 1.1336 (less than t_{cr}), and so INSIGNIFICANT

t_3 = : 6.78811 (not less than t_{cr}), so SIGNIFICANT

$t_{12} = 3.01394$ (not less than t_{cr}), so SIGNIFICANT

$t_{13} = 1.72289$ (not less than t_{cr}), so SIGNIFICANT

$t_{23} = : 1.52497$ (less than t_{cr}), and so INSIGNIFICANT

$t_{123} = 1.57894$ (less than t_{cr}), and so INSIGNIFICANT

The accepted regression terms before the conversion :

0.151737	(TAKEN)
0.00235	X1 (NOT TAKEN)
-0.001575	X2 (NOT TAKEN)
0.00943124	X3 (TAKEN)
-0.0041875	X1 X2 (TAKEN)
0.00239375	X1 X3 (TAKEN)
-0.00211875	X2 X3 (NOT TAKEN)
0.00219375	X1 X2 X3 (NOT TAKEN)

Mathematical model in transformed variables is :

$$y = 0.151737 + 0.00943124X3 - 0.0041875X1X2 + 0.00239375X1X3 \tag{4.24}$$

Mathematical model in real variables is :

$$Y = 0.163446 - 0.00167917A + 0.0161917C - 0.00799834D - 0.000223333AC + 0.000127667AD \tag{4.25}$$

Examination of the adequateness of the model :

The estimated averages of the responses are shown in Table 4.12:

The Variance of adequateness : $s^2_{ad} = 0.000138438 < s^2\{y\} = 6.17719^{e-05}$

$f_{ad} = n - m = 8 - 4 = 4$

F-Criterion : $F = 2.24112 < F_{cr}$ (5.14) shows the adequateness of the model.

Table 4.12 - Estimated averages of the response (damping ratio).

Points of matrix-u	$\bar{y}_u$	$\hat{y}_u$	$(\bar{y}_u - \hat{y}_u)^2$
1	0.135425	0.140513	**0.000026**
2	0.1481	0.1441	**0.0**
3	0.149275	0.148888	**0.0**
4	0.136425	0.135725	**0.0**
5	0.158125	0.154587	**0.000013**
6	0.1716	0.16775	**0.00001**
7	0.154725	0.162962	**0.000068**
8	0.160225	0.159375	**0.000001**

4.12 Analysis of the results.

The objective of using DOE is to ascertain the affect of RTM process parameters on the five mechanical properties chosen for examination. All the graphs obtained for the five mechanical properties are appended as Appendix (D). In the present scenario a regression equation is used to reflect the phenomenon under study. Initially the significant factors affecting the response are identified, then all the significant factors are included in the regression equation. From the regression, by substituting the intervals of variation of the significant factors, we obtain the final regression equation in the true values of variables, which is called the parameter of optimization.

Similar to the sieve design results, the DOE results have also shown that different factors have different effects on the various mechanical properties under study. In comparison to the shear strength of the laminates which were studied in the sieve design analysis, the shear strength of the laminates in the DOE analysis have shown a marked improvement. Laminates have also shown a marked improvement in flexural strength. The damping ratio has also decreased appreciably, from a high of 0.3965 in the case of sieve design analysis, to a high of 0.1716 in the case of DOE analysis. In the case of the glass transition temperature, higher values of glass transition (Table 3.7) were possible when factor D (post curing temperature), is placed at its upper interval (95 °C). This is in tune with the earlier sieve analysis studies where higher temperatures were abetting the values of glass transition temperatures. Flexural storage modulus values have averaged without much variation. The less discrepancies in the results obtained from both analysis probably arise because this mechanical property is either independent, or less sensitive to the changes in the levels of process parameters.

The increase in strength of laminates studied under DOE analysis can be attributed to the re-arrangement of the factor intervals, and also to the increase in the no. of preform layers. Chopped strand matt was also one of the main reasons for the reduced mechanical properties in the case of sieve design analysis. After the elimination of chopped strand matt, as a factor, the variance in the mechanical properties has been reduced, and has considerably averaged out. This is evident in the case of all the mechanical properties (Tables 3.3, 3.5, 3.7, 3.9, and 3.11).

The increase in the strength can also be attributed to the lower injection pressures, and also to the higher levels of vacuum. Maintaining the vacuum level at 25 mm Hg, has considerably decreased the percentage of the voids present in the laminate. This is a marked improvement over the percentage of the voids present at the sieve design analysis stage. The only visible void content was at the injection side of the laminate, and it could be due to the bubbles present in the resin. The air bubbles in the resin could have been produced by the mechanical action of the injection pump, or these might be the trapped air present in the resin while it is transferred into the RTM machine. Also lowering the injection pressure has provided enough time for resin to penetrate into the fibers. This was evident by the difference in the cycle times noticed in the two experimental phases. Cycle times were very short in the sieve analysis stage, at around 2 minutes. In the case of DOE analysis the cycle times were longer, at around 3 to 4 minutes.

The reduction in the damping ratios is one of the interesting observations of the DOE analysis. Damping ratio is the ratio of the energy dissipated to the energy stored in the material under load. This energy dissipation may be caused by viscoelastic dissipation of the resin, friction at fiber-resin interface, and high level of voids. Higher curing temperature increases the degree of cross links in the resin, and impart extra hardness to the resin. This also results in higher glass transition temperatures, and reduced viscoelastic dissipation of the resin. The glass transition temperature values seen in the sieve analysis are quite high, this could be due to the curing temperatures used in the sieve analysis that are above the levels specified by the resin manufacturer. The void content in the laminates produced during the sieve analysis is also high, and due to higher injection pressures, the wetting of the fiber was improper, thus providing a weak bond between the fiber and the resin. So, if the interface bonding is poor, and the void level is high, the composite exhibits a high damping ratio. This was the cause of the higher damping ratios during the sieve analysis.

In the case of DOE analysis, the post curing temperatures are in the range specified by the resin manufacturer. So, the glass transition temperature values are less compared to the values of the sieve analysis. The void content is also reduced considerably because of high level of vacuum. Because of the lower injection pressures, there was sufficient time for the resin to cause a good wetting, thus improving the bond with the fiber. So, in the DOE analysis, not only the resin is cured properly, but also the quality of the composite is improved in terms of good interface bonding, and lessened void content. This is the cause of the reduced damping ratios.

Analyzing the affect of various factors on the five mechanical properties. In the case of shear strength, the significant factors are injection pressure (A), pre-molding temperature (C), and post curing temperature (D). The interaction effects of factors AC, AD, CD, and ACD, are also significant. All the main factors A, C, and D are contributing for an increase in the response (Eq 4.13). Except for the interaction effect of factor ACD, all the other interaction effects are contributing for a decrease of the response.

In the case of flexural strength, all the main factors, A, C, and D have significant affect on the response. Factor AD, is also significant as an interaction factor. All the main factors are contributing for an increase in the response, but the interaction factor has a negative effect thus contributing to a decrease in the response.

Analysis of the glass transition temperature reveals that all the main factors, and all interaction factors in the study are significant. All the main factors A, C, and D have a negative influence on the response (Eq 4.21). Except for interaction factors AC, and AD, the other interaction factor has a negative influence on the response.

In the analysis concerning the flexural storage modulus, all the factors are significant. All the main factors are contributing for an increase in the response, whereas the interaction factors, except the interaction factor ACD, have a negative affect on the response.

In the case of the damping ratio, Factors A, C, D, AC, and AD are the main and interaction factors that are having influence on the response (Eq. 4.25). Factors A, and D are contributing for a decrease in the response. Whereas main factor C, is having a positive influence on the response. As an interaction factor, factor AC, is having a negative influence whereas factor AD, is having a positive influence, on the response.

4.13 Conclusion

The rearrangement of the ranges of the factors that were significant after the sieve analysis, have shown marked improvement in the mechanical properties of the laminates. The void content in the laminates has been significantly reduced thus improving in the mechanical properties. All the three factors and their interaction effects are significant for majority of the mechanical properties. The ranges at which they have an influence on the mechanical properties also significantly varies from property to property. Flexural storage modulus is the only mechanical property that is insensitive to changes in the ranges of the factors.

CHAPTER 5

Conclusion and future work

Resin Transfer Molding Process (RTM) is one of the manufacturing techniques that has become effective for production of fiber reinforced composite components. This manufacturing process like many other process involves a large number of variables, consisting of both material, and processing parameters. In the present study ten independent and controllable RTM process variables are identified and systematically studied to investigate the effect of these factors on the mechanical properties of the laminates produced through the RTM process. This is for the first time that such a comprehensive list of factors covering the RTM process are studied.

In order to identify the significance of the factors, statistical experimental design analysis is used as a tool. The statistical experiment is conducted in two phases. In the first phase of the experiment all the ten factors were studied using for the first time a sieve design analysis. A design matrix, with factors at two levels, and 16 runs is constructed using the sieve methodology. Laminates were made using the levels specified in the matrix. Specimens, with sizes specified by the ASTM standards, were cut and three different types of tests were conducted. From the data of the tests, five mechanical properties, namely, flexural strength, shear strength, flexural storage modulus, glass transition temperature, and damping ratio, were studied. All the results were analyzed using the scatter diagrams. Student t-criterion is used to test the significance of the factors.

From the sieve analysis it was apparent that various factors affect the mechanical factors differently. Chopped stranded mat has a negative influence on almost all of the mechanical properties. This was due to swaying away of the chopped fibers towards the outlet side of the mold. This is possibly due to either the high injection pressure, which could have pushed the fiber, or it might be due to the vacuum employed at the outlet port. One more interesting observation is the increase in the flexural strength of the laminate with the increase in the number of the layers. This could be due to the usage of the vacuum to evacuate the mold, which paved the way for better wetting of the fiber by the resin, thus increasing the strength. Pre-mold temperature has positive influence on the response, thus increasing the properties of the laminate. This is due to the fact that, higher pre-mold temperature has decreased the resin viscosity, thus allowing for a better penetration and wetting of the fiber. Flexural storage modulus is the mechanical property which was insensitive to the changes in the levels of the factors. Damping ratio is influenced by hydrocheck pressure, and resin to catalyst ratio. Post curing temperature has a high influence on the glass transition temperature. Both the temperatures are in fact influencing the glass transition temperature. So, if glass transition temperature is the desired property, then selection concentration should be on post-curing temperature and pre-mold temperature.

From the sieve analysis, only three factors, injection pressure, pre-mold temperature, and post curing temperature were selected for further analysis. These were the factors that determined the majority of the mechanical properties. Their influence was observed either as main factors, or as interaction factors. All the remaining factors were kept at levels at which they had a positive influence on the response. For the second phase of the experiment, vacuum was placed at a higher level. In the laminates produced by the sieve experiments, there was a high concentration of voids at the outlet port of the mold. This could be due to the improper evacuation of the mold, i.e., the lower vacuum levels used in the first phase of the experiment. Since the increase in the number of the layers had a positive influence, the number of layers were increased for the second phase of the experiment.

In the second phase of the study, all the factors that proved to be significant in the sieve experiment, are re-experimented with changed ranges. Design Of Experiments (DOE), is used as a tool to study the significance of the factors. Similar to the sieve design analysis, the analysis of DOE had showed that various factors have different affect on different mechanical properties. Also the ranges at which they produce an optimum response, differed from property to property. The mechanical properties have shown a marked improvement.

Shear strength, and flexural strength have improved. Higher vacuum levels had decreased the void content, thus aided for the improvement of the mechanical properties. Lower injection pressures decreased the rate of flow of the resin in the mold, and thus improved the fiber wetting and paved the way for a good bond between the fiber and resin. The damping ratios in the DOE analysis have also dropped down, because of the above reasons, as desired for a good laminate. DOE analysis has also shown that the glass transition temperature is a property that is mainly dependent on the post curing temperature. The lower values of the post curing temperature have decreased the glass transition temperature, and higher glass transition values are observed when the post curing temperature is at a higher level.

The main significance of this study is the derivation of the regression equations to ascertain the optimum response of the mechanical properties. Through the DOE analysis, optimization equations are found for the five mechanical properties under study. All the mechanical properties have various responses to the RTM process parameters, and to their interactions. Depending upon the desired property from the RTM laminates, any manufacturer, for a better laminate, can use the regression equations to optimize the desired mechanical properties.

For future work, the levels of the significant factors are to be rearranged, to check for further improvement of response, i.e., the mechanical properties. Further study can be done by choosing resin as an independent factor. The effect of changes in the resin flow, and viscosity on the mechanical properties would be an interesting paradigm for further study.

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APPENDIX - A

SEIVE ANALYSIS - SCATTER GRAPHS

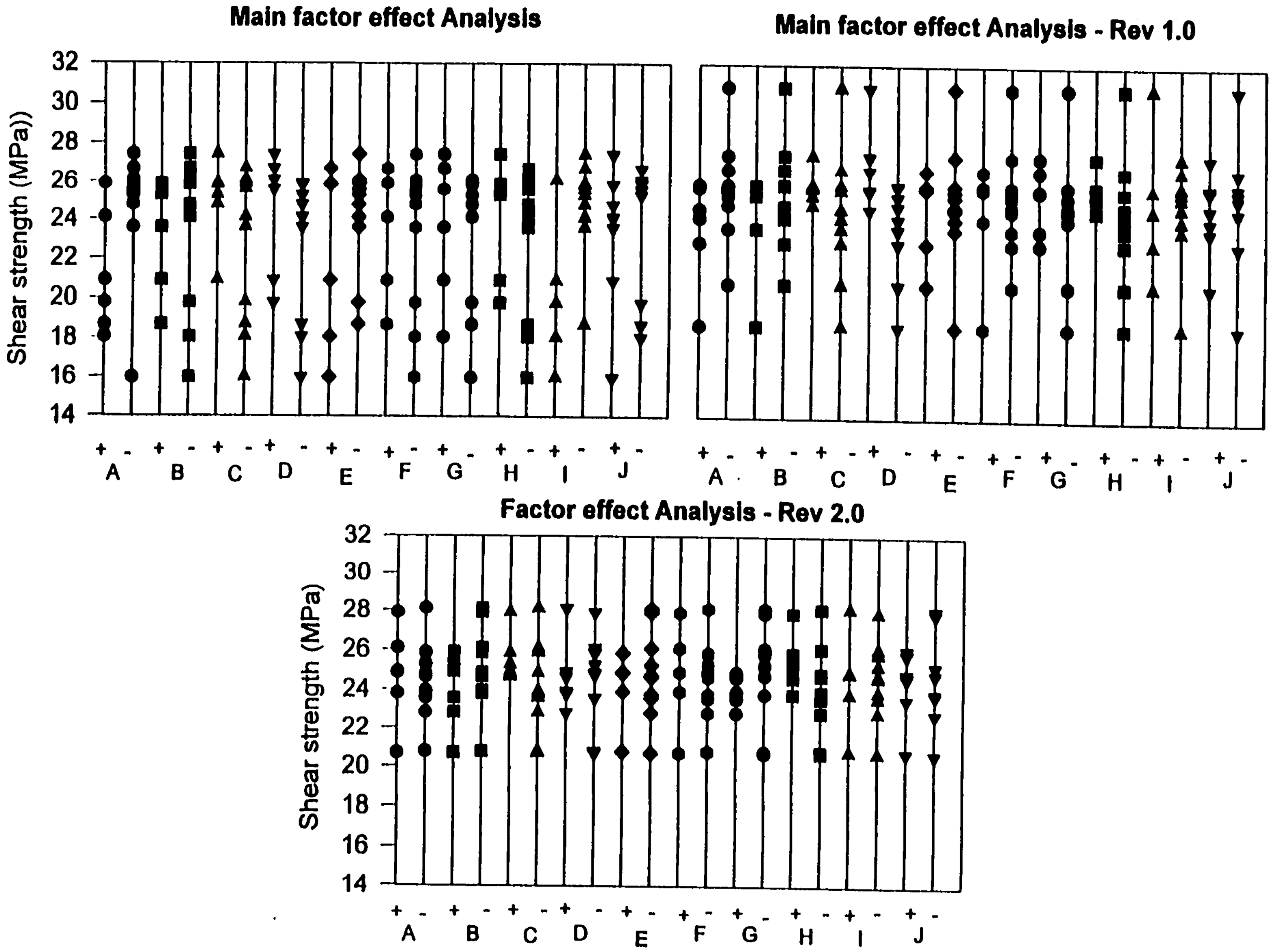
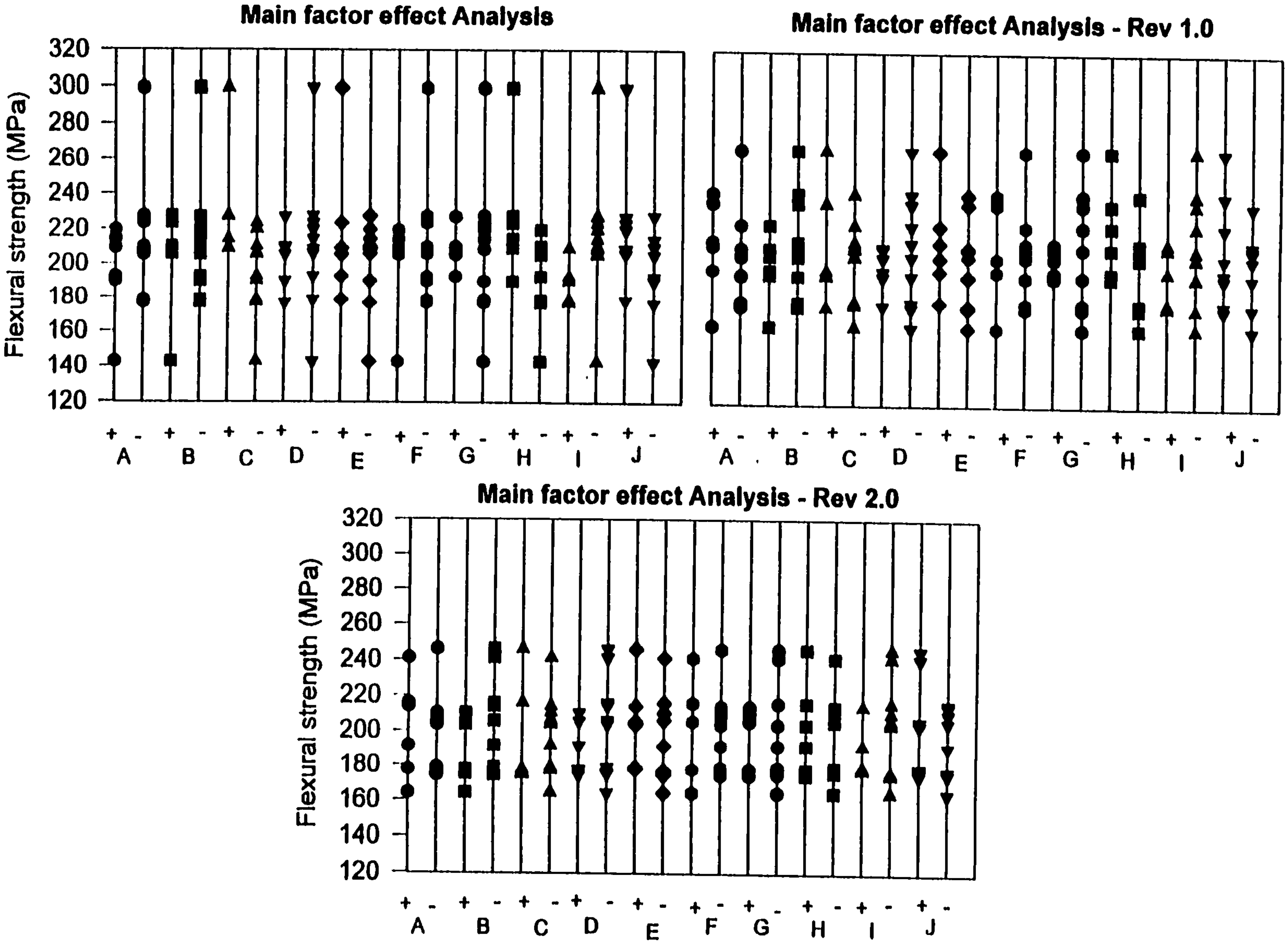


Fig. A.1 - Shear strength main factor analysis.



F.g. A.2 - Flexural strength main factor nalysis.

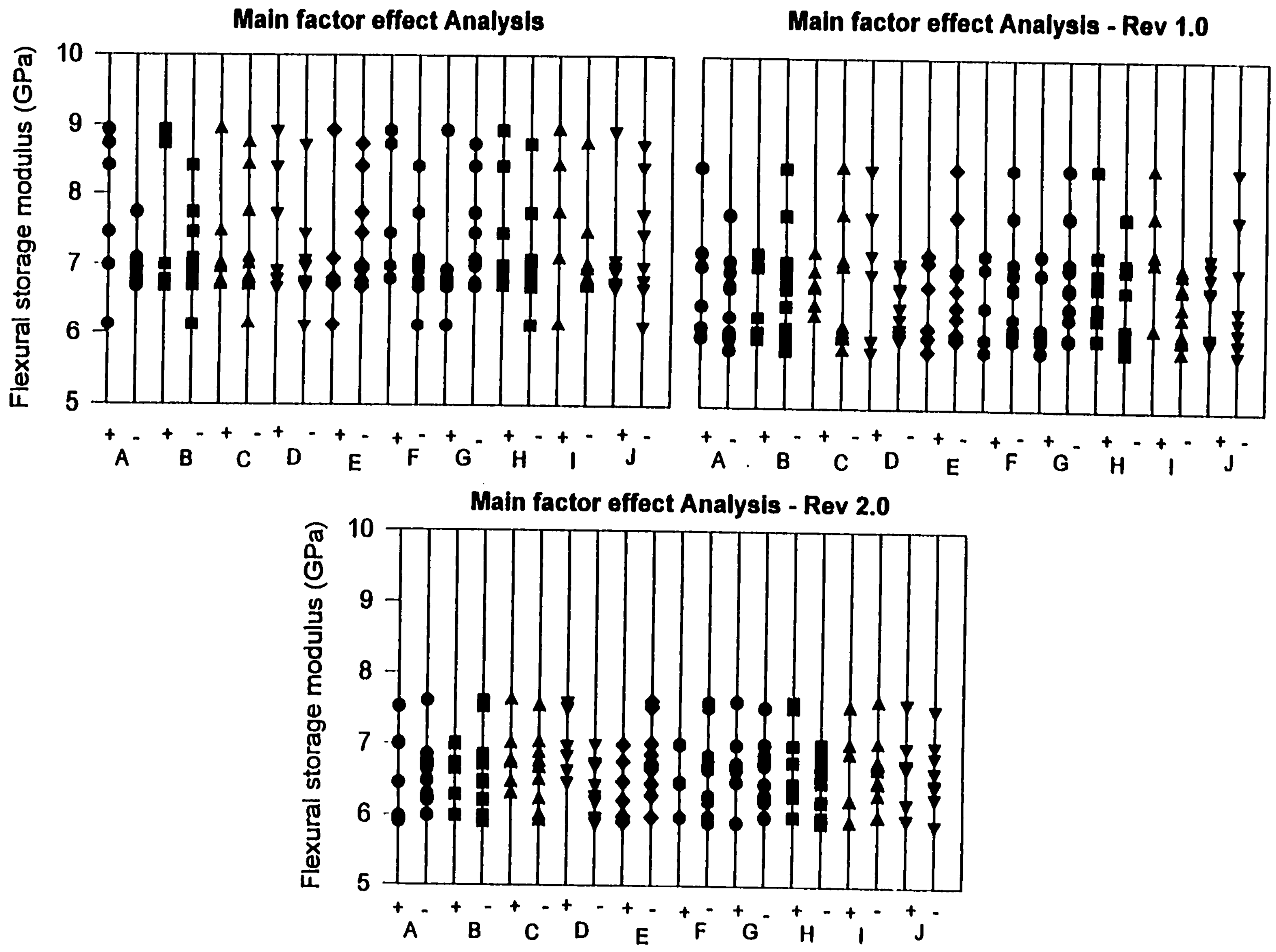


Fig. A.3 Flexural storage modulus main factor analysis.

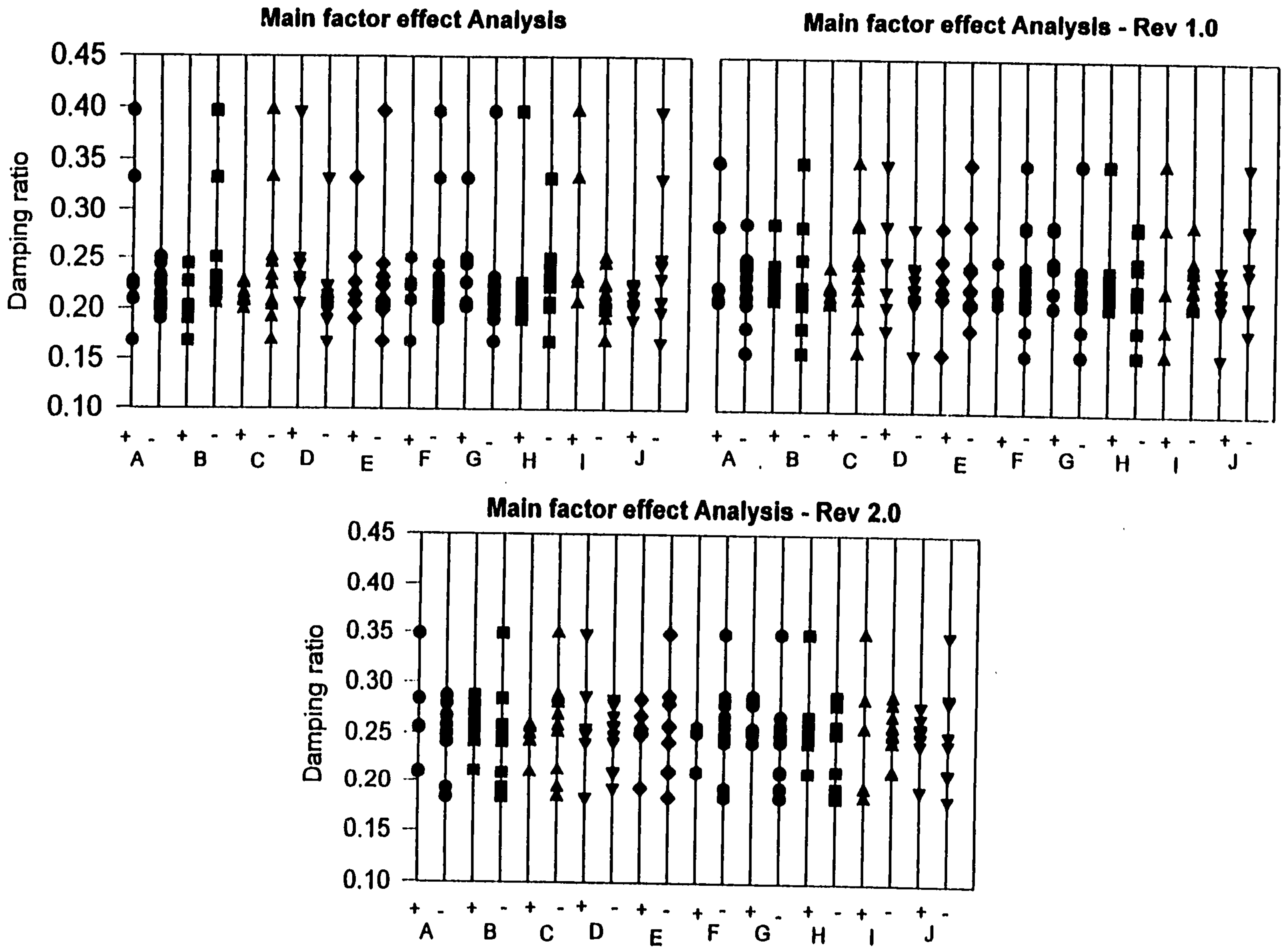
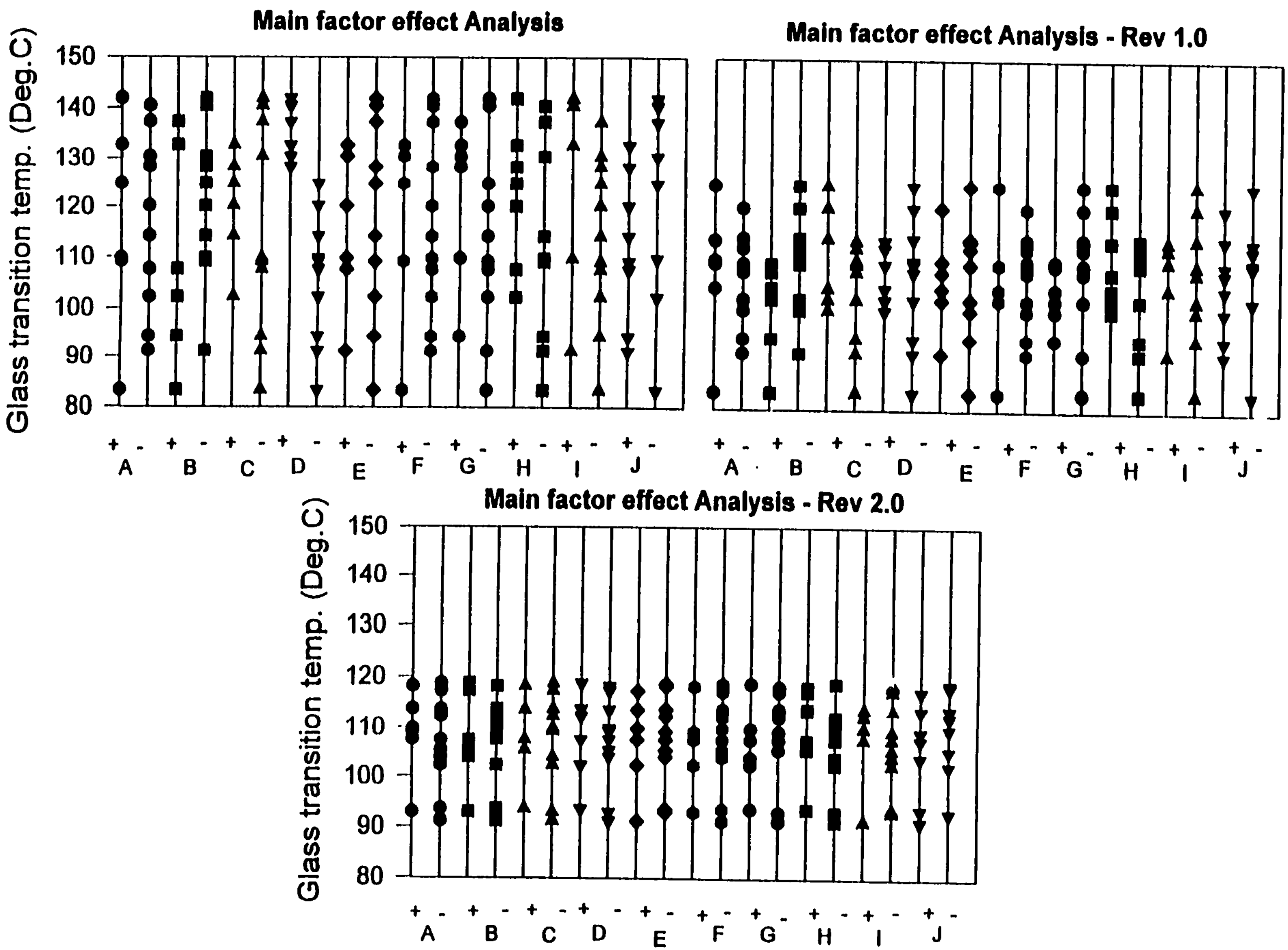


Fig. A.4. - Damping ration main factor analysis.



Main factor effect Analysis

Main factor effect Analysis - Rev 1.0

Main factor effect Analysis - Rev 2.0

Fig. A.5. - Glass transition temperature main factor analysis.

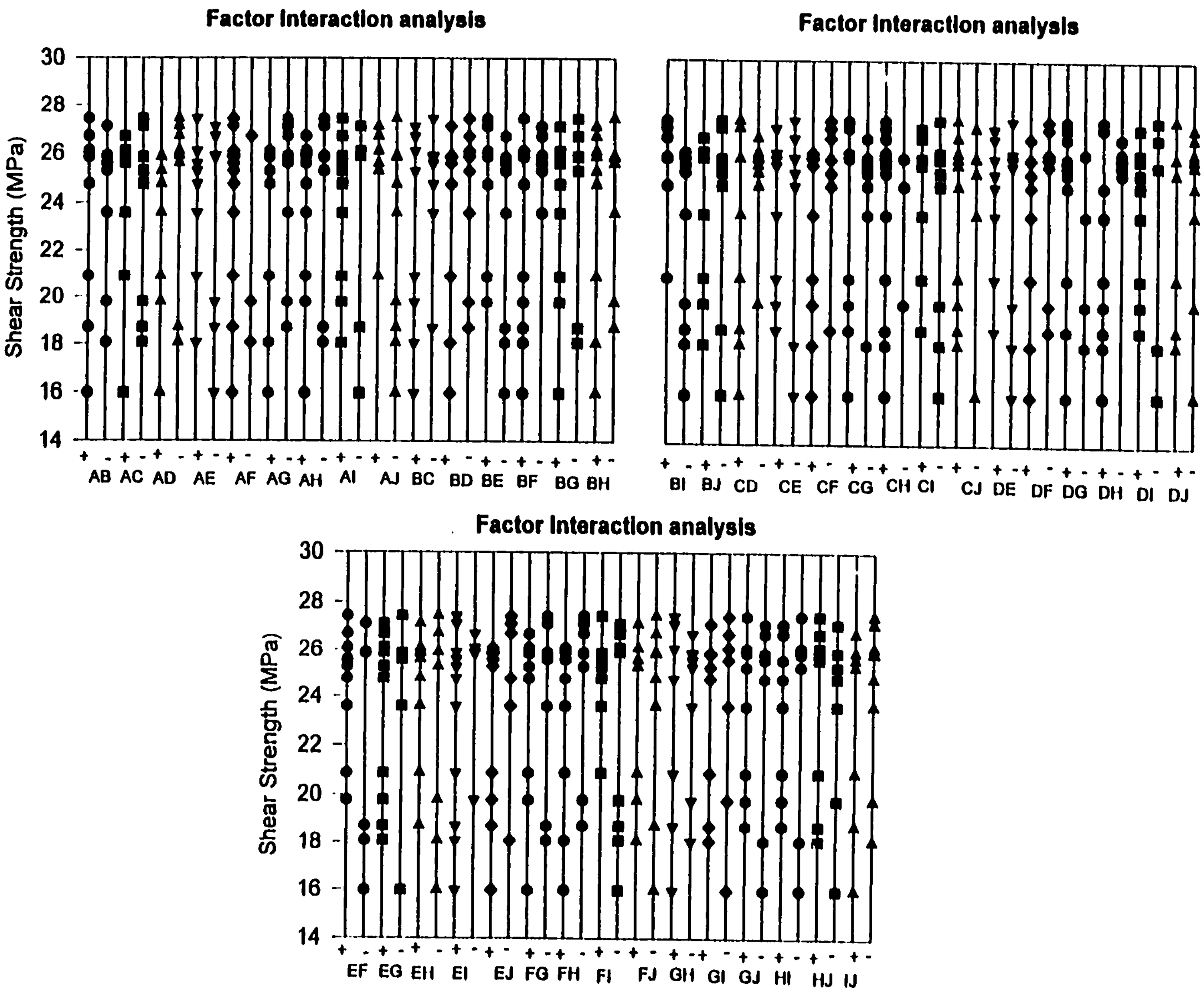


Fig. A.6. - Shear strength factor interaction analysis.

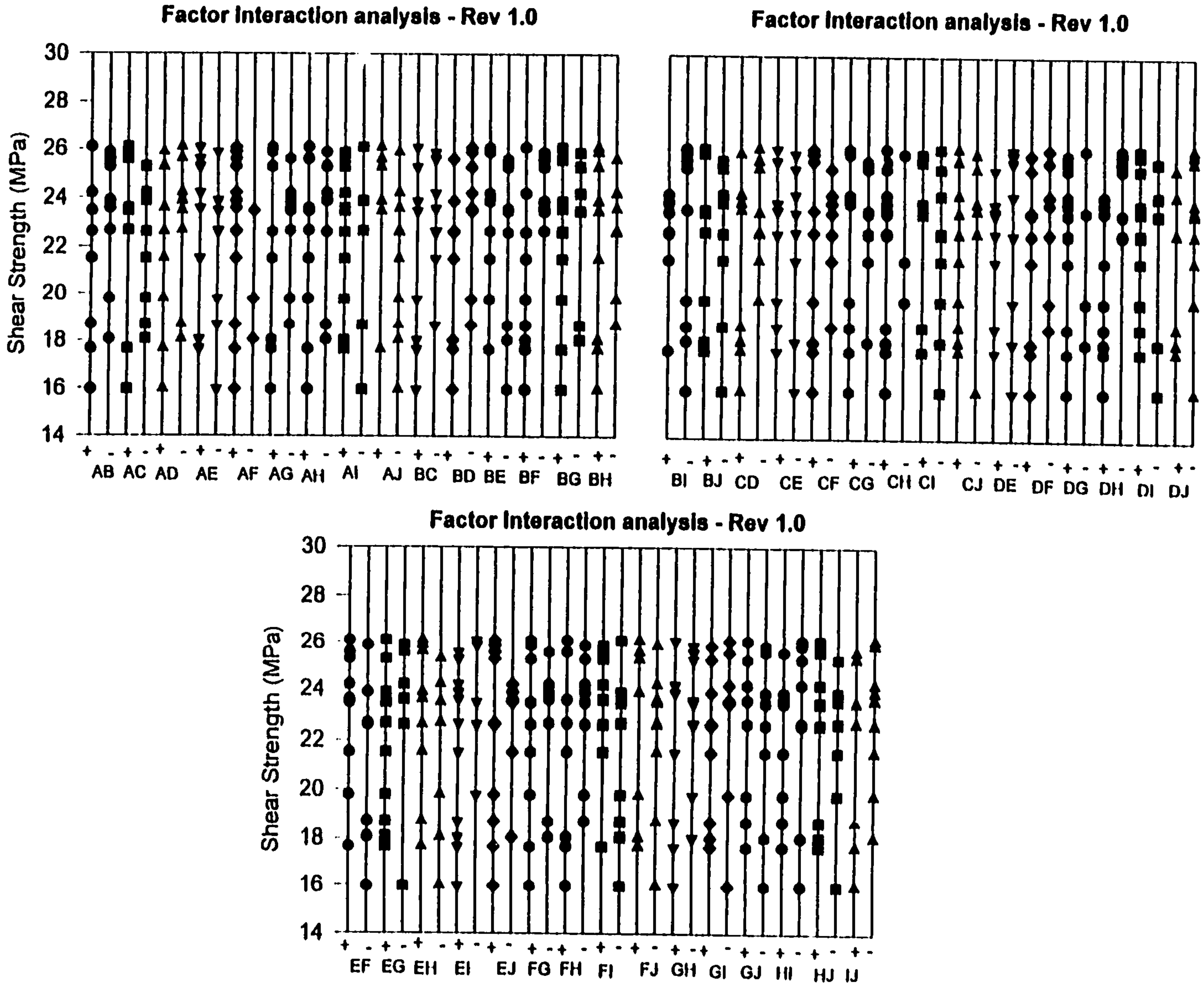
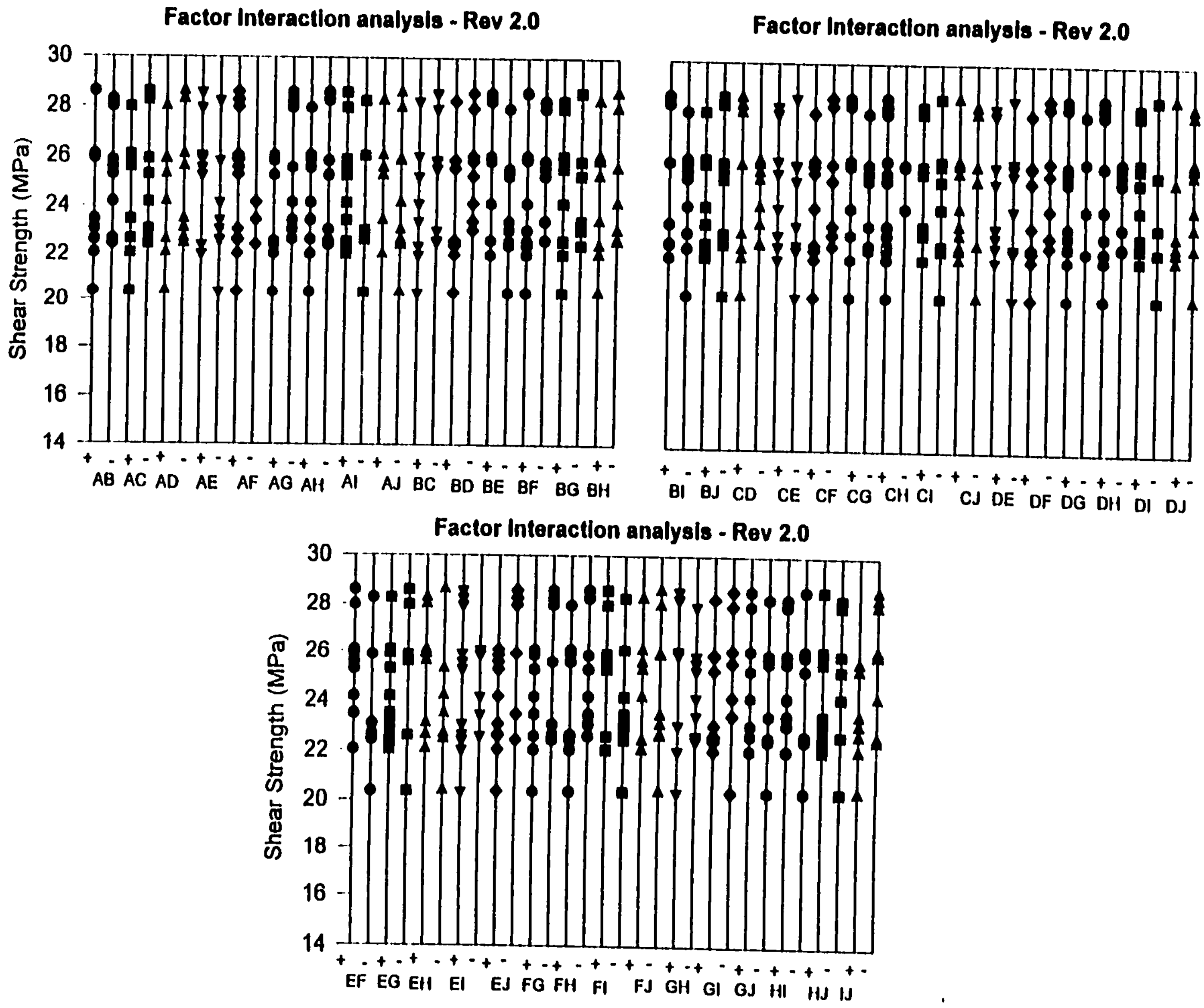


Fig. A.6. - Shear strength factor interaction anlysis.



F.g. A.6. - Shear strength factor interaction analysis.

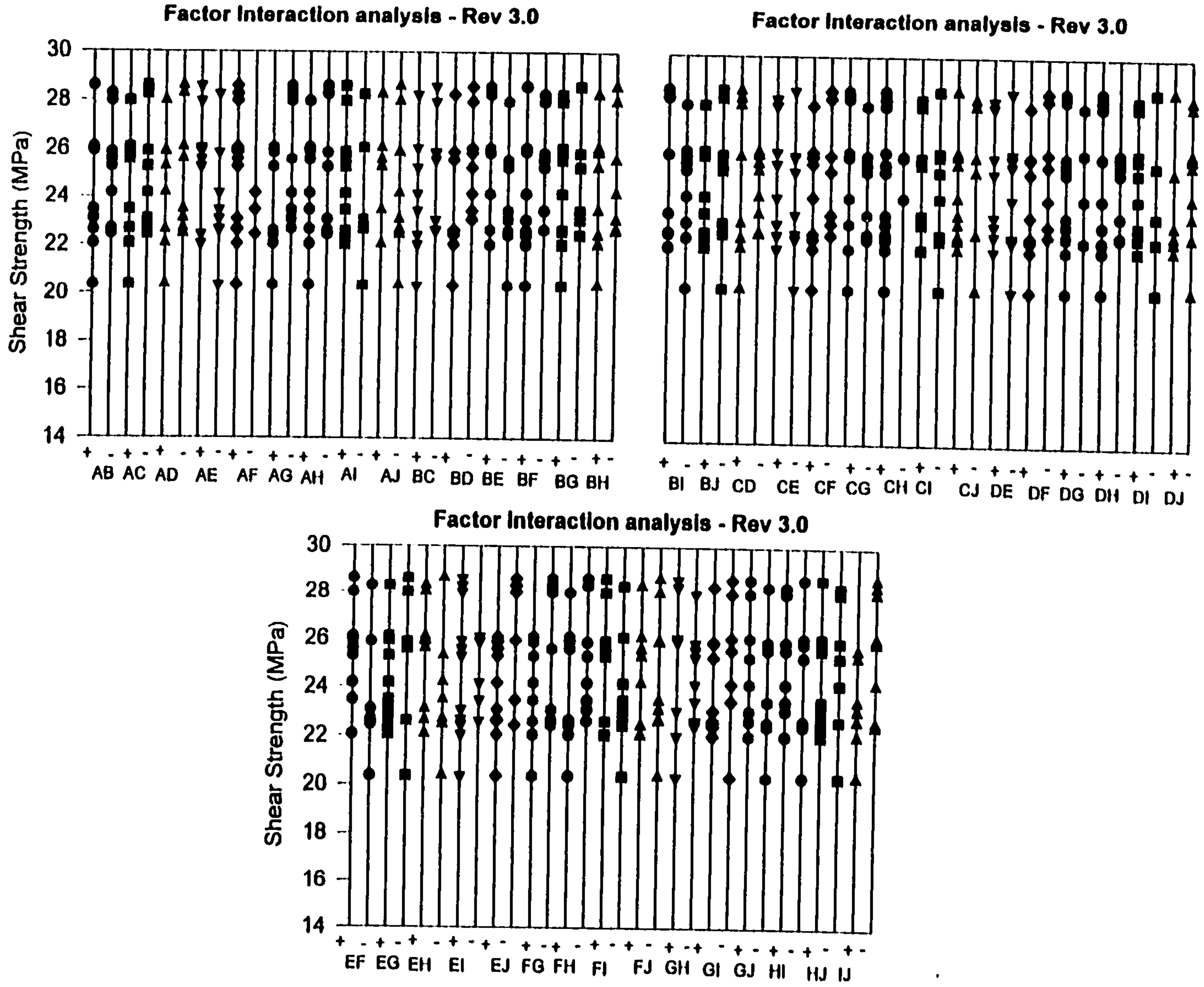


Fig. A.6. -Shear strength factor interaction analysis.

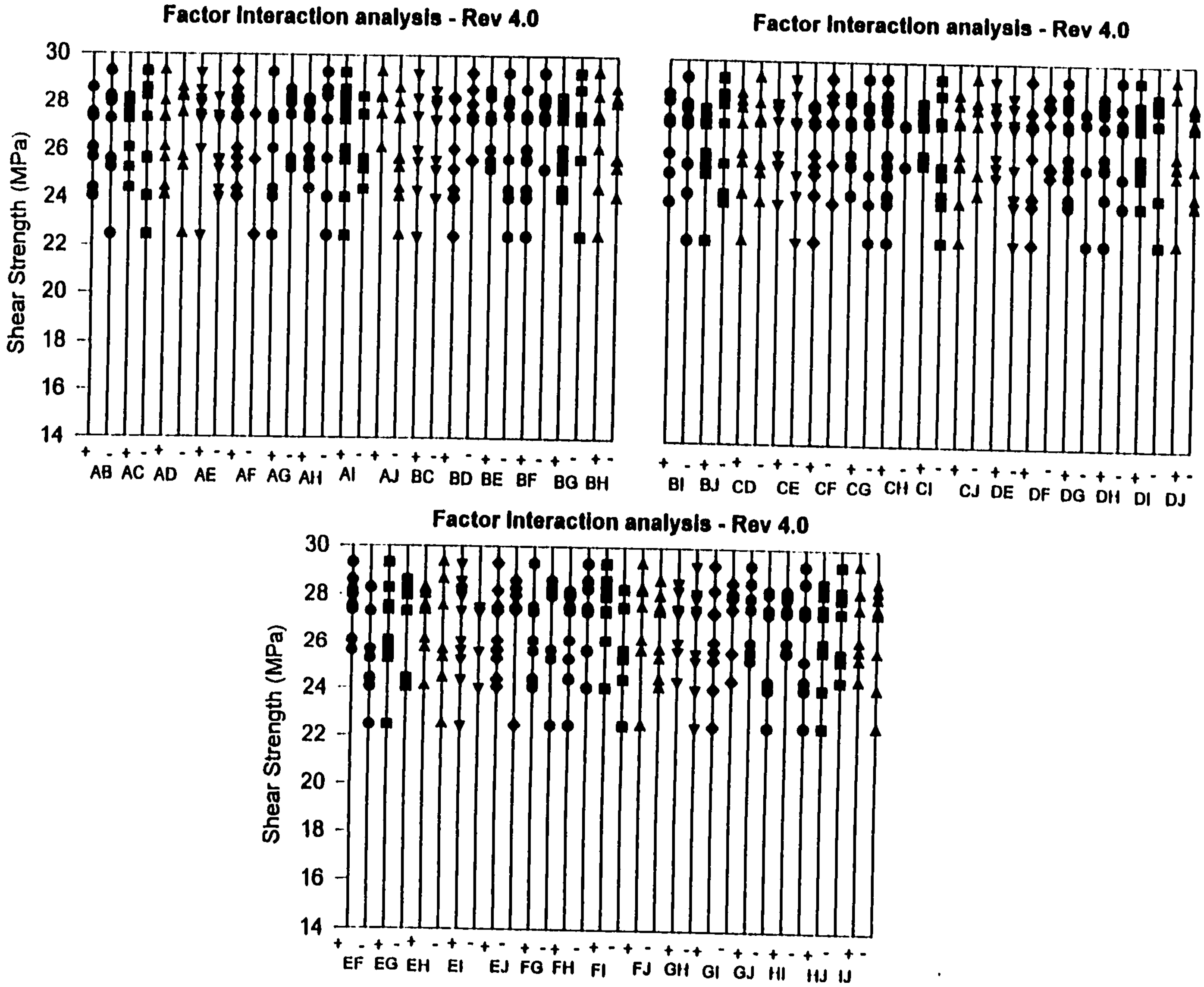


Fig. A.6. Shear strength factor interaction analysis.

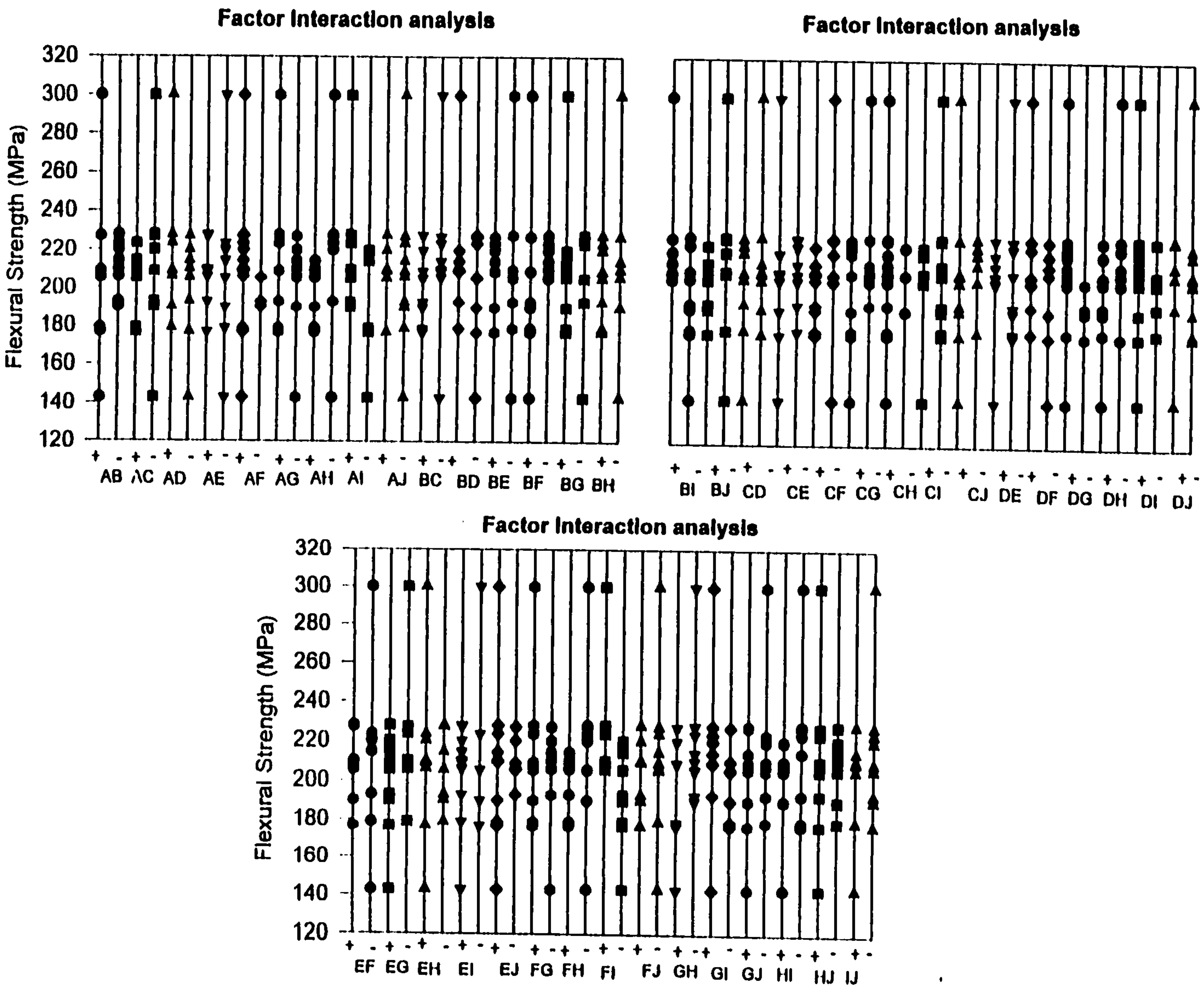
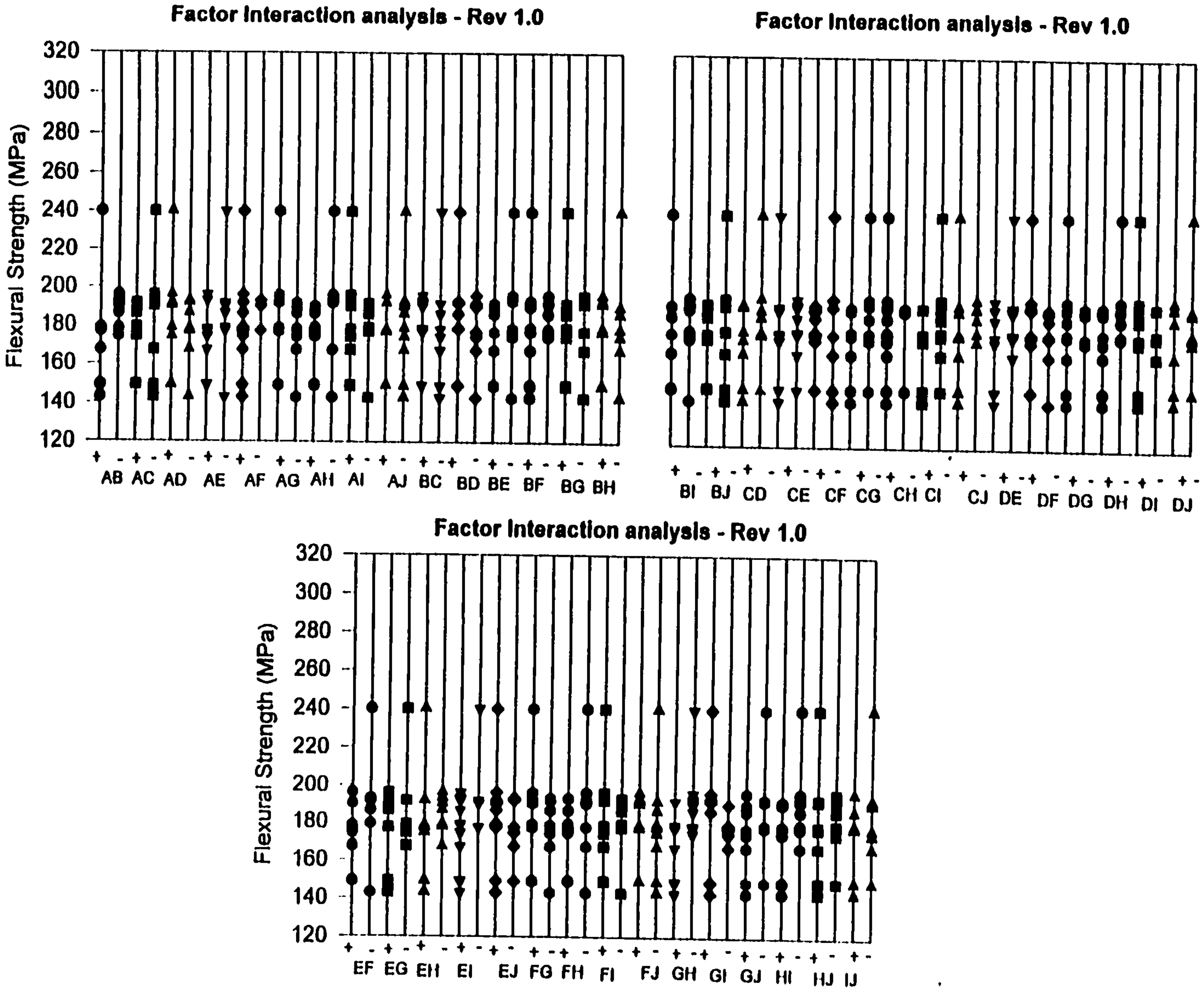


Fig.A.7. - Flexural strength factor interaction analysis.


Factor Interaction analysis - Rev 1.0

Factor Interaction analysis - Rev 1.0

Factor Interaction analysis - Rev 1.0

Fig.A.7. - Flexural strength factor interacion analysis.

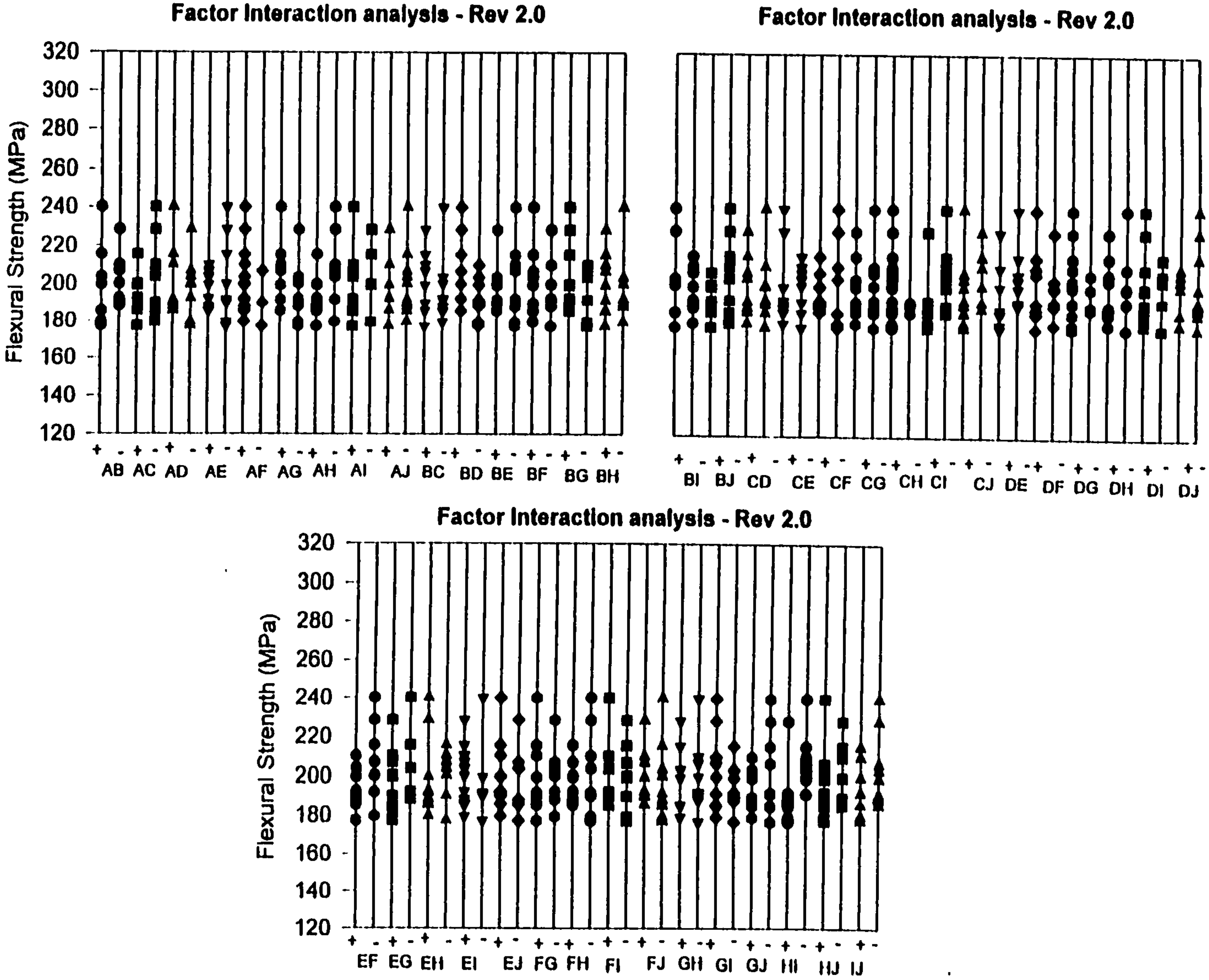
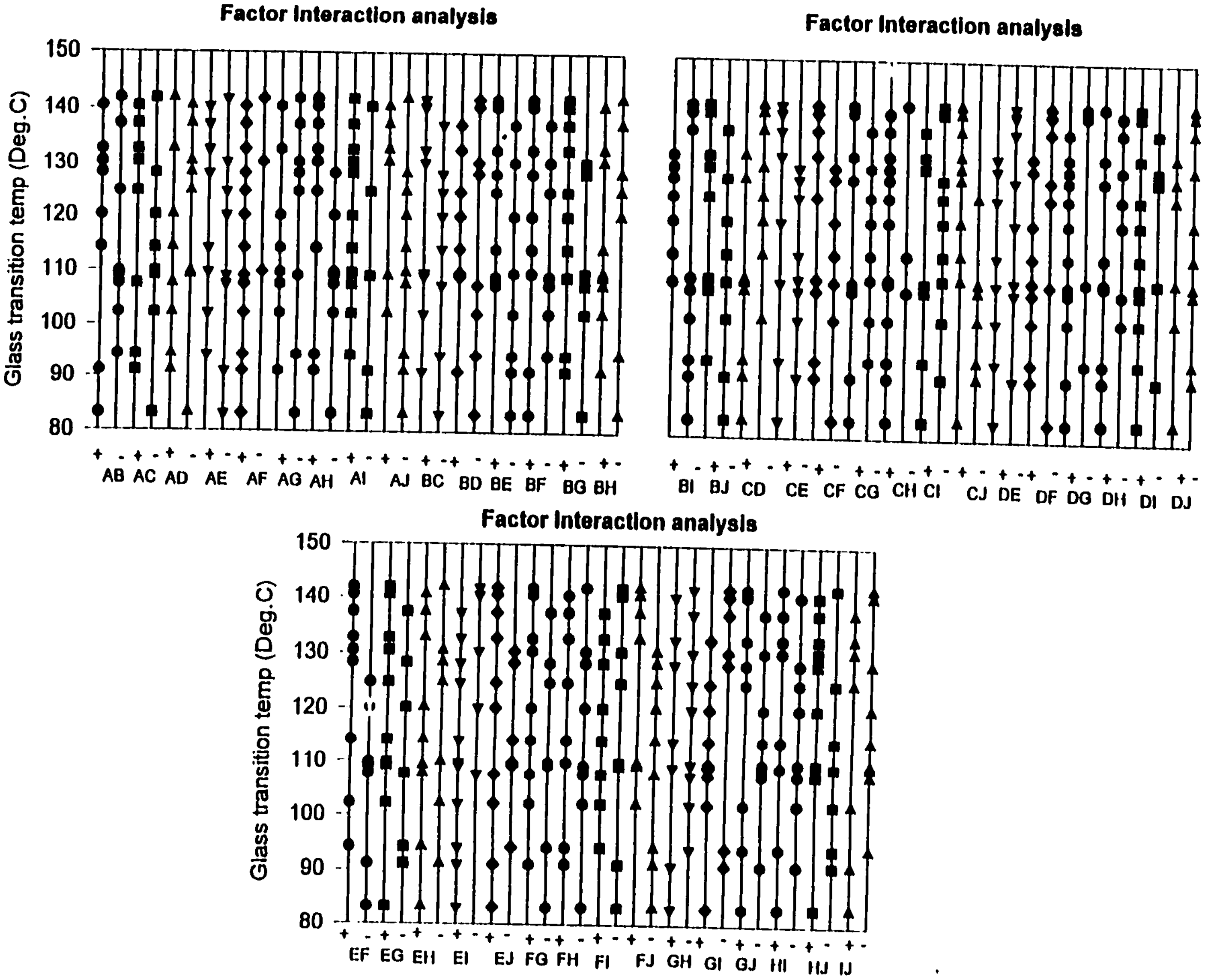


Fig.A.7 - Flexural strength factor interaction analysis.



Factor Interaction analysis

Factor Interaction analysis

Factor Interaction analysis

F.g. A.8. - Glass transition temperature factor interaction analysis.

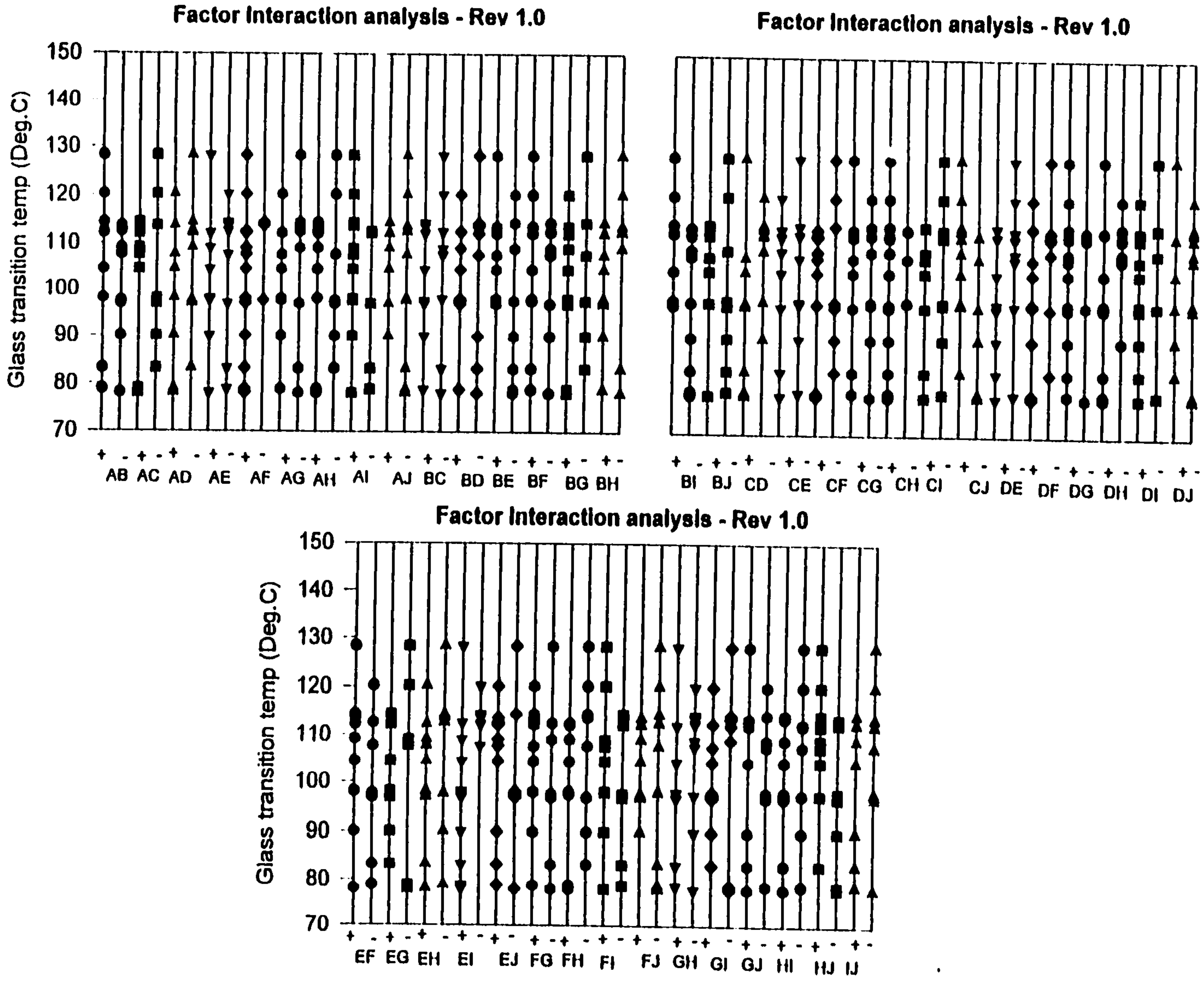
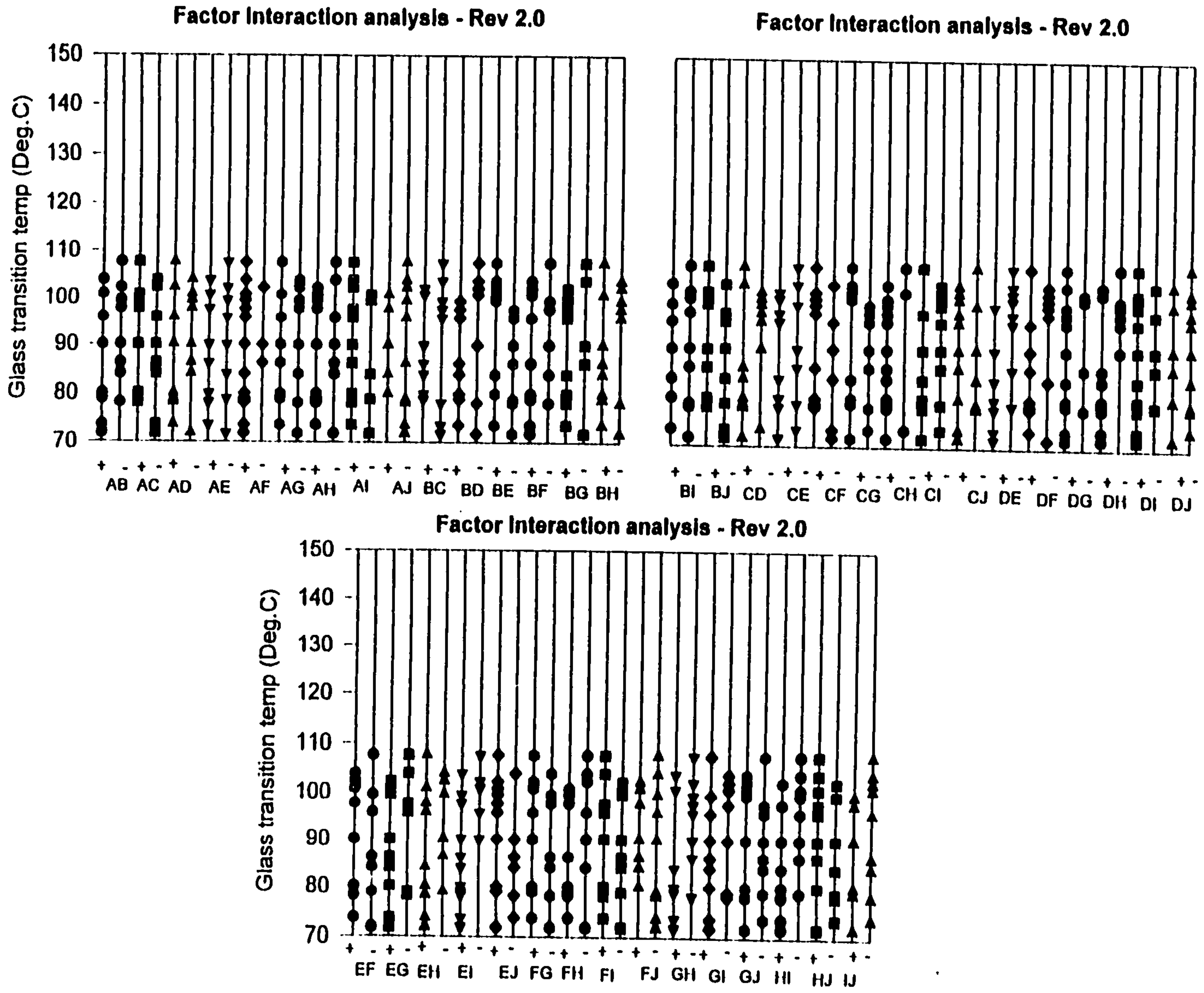


Fig. A.8. Glass transition temperature factor interaction analysis.



Factor Interaction analysis - Rev 2.0

Factor Interaction analysis - Rev 2.0

Factor Interaction analysis - Rev 2.0

Fig. A.8. Glass transition temperture factor interaction nalysis.

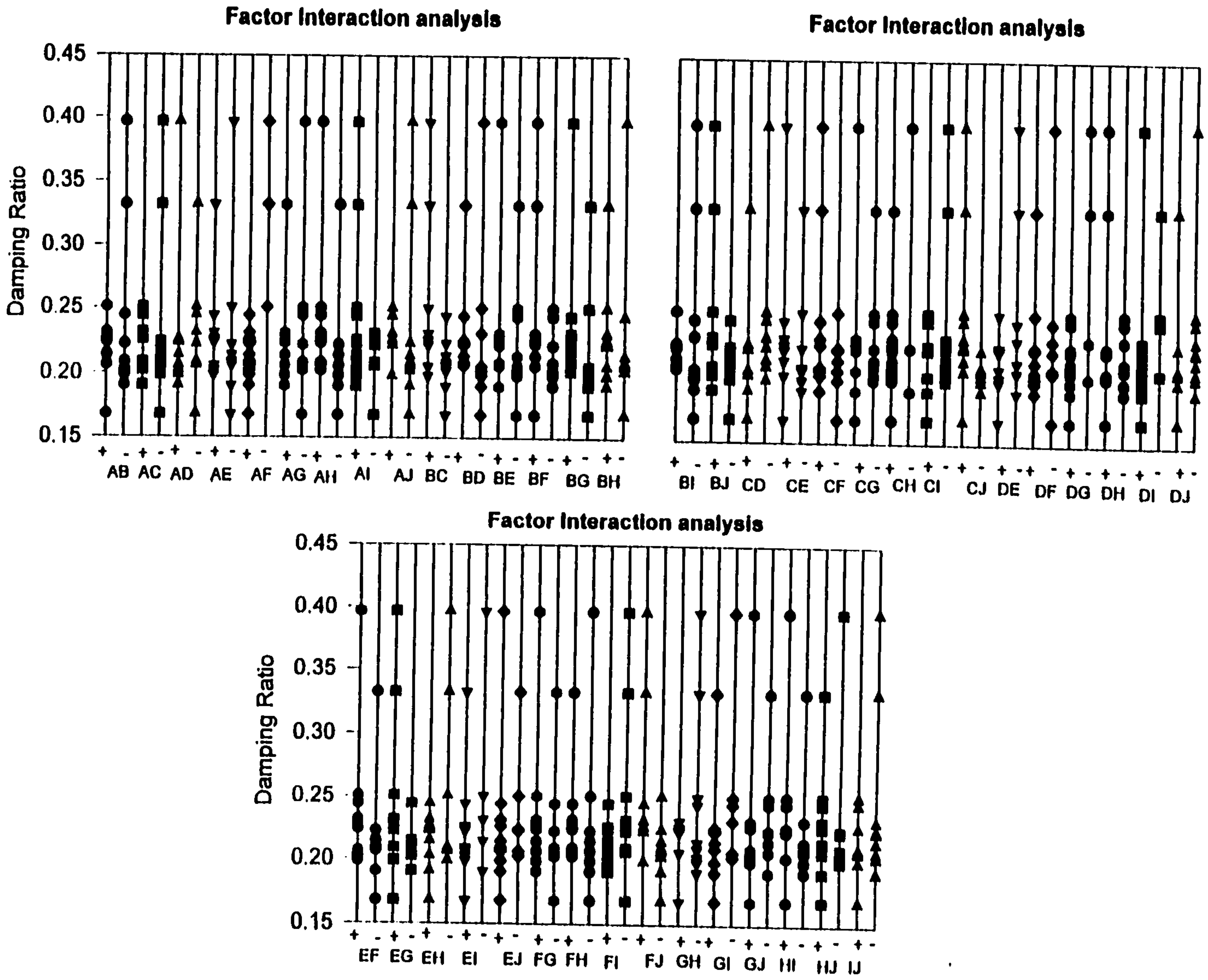


Fig. A.9. - Damping ration factor interaction analysis.

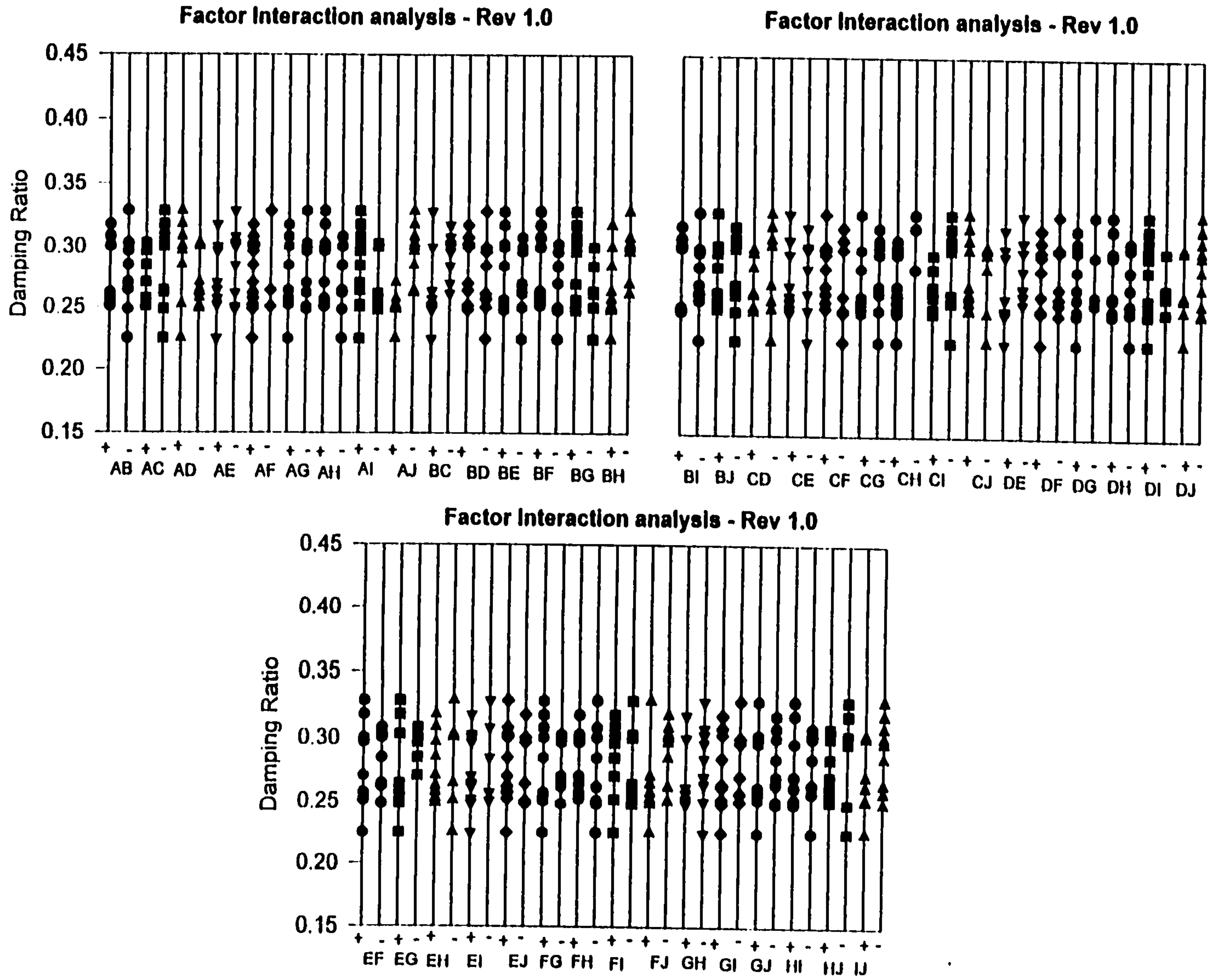
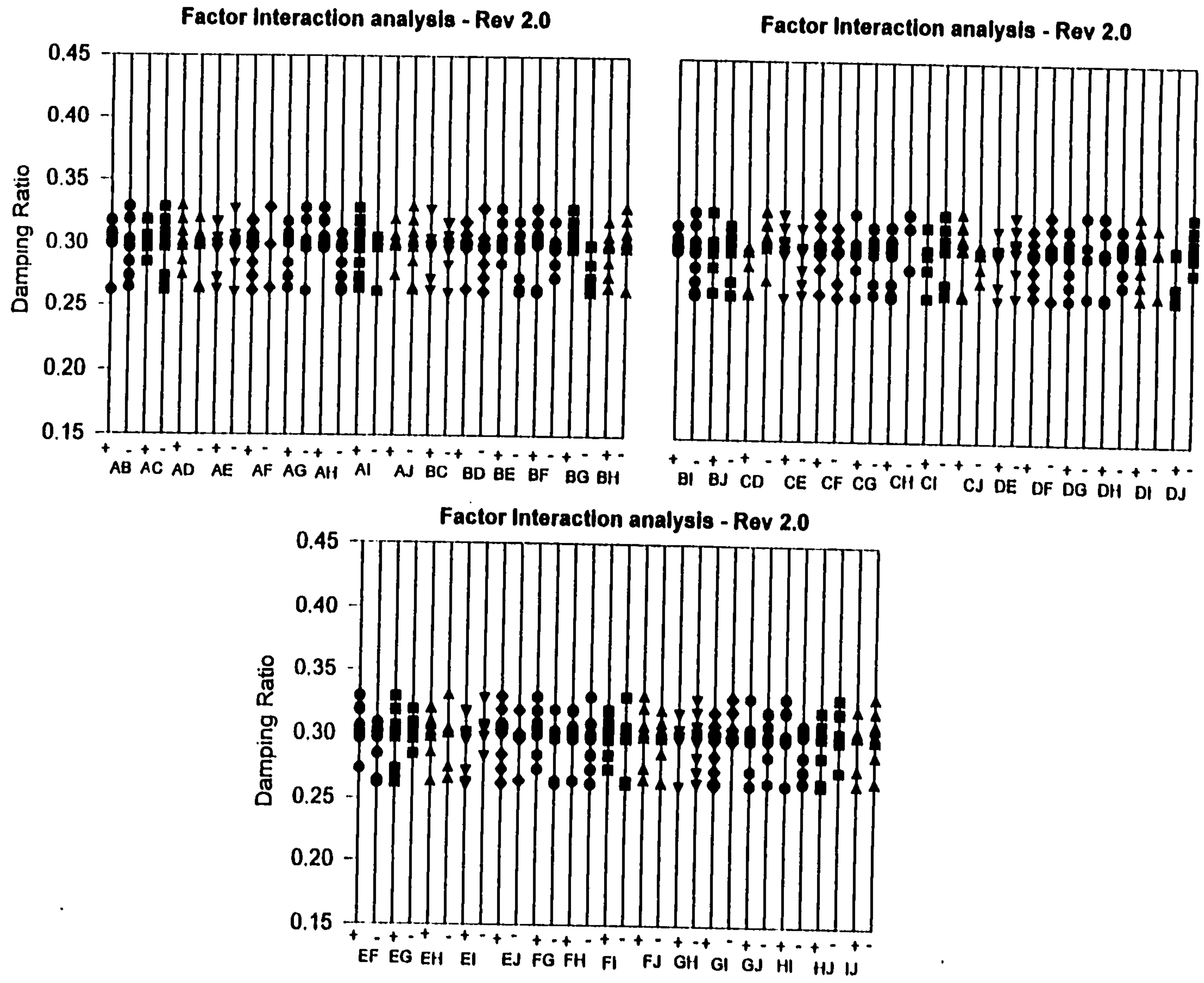


Fig. A.9. - Damping ration factor interaction analysis.



<u>F.gA.9.</u> <u>-</u> <u>Damping</u> <u>ration</u> <u>factor</u> <u>interaction</u> <u>analysis.</u>

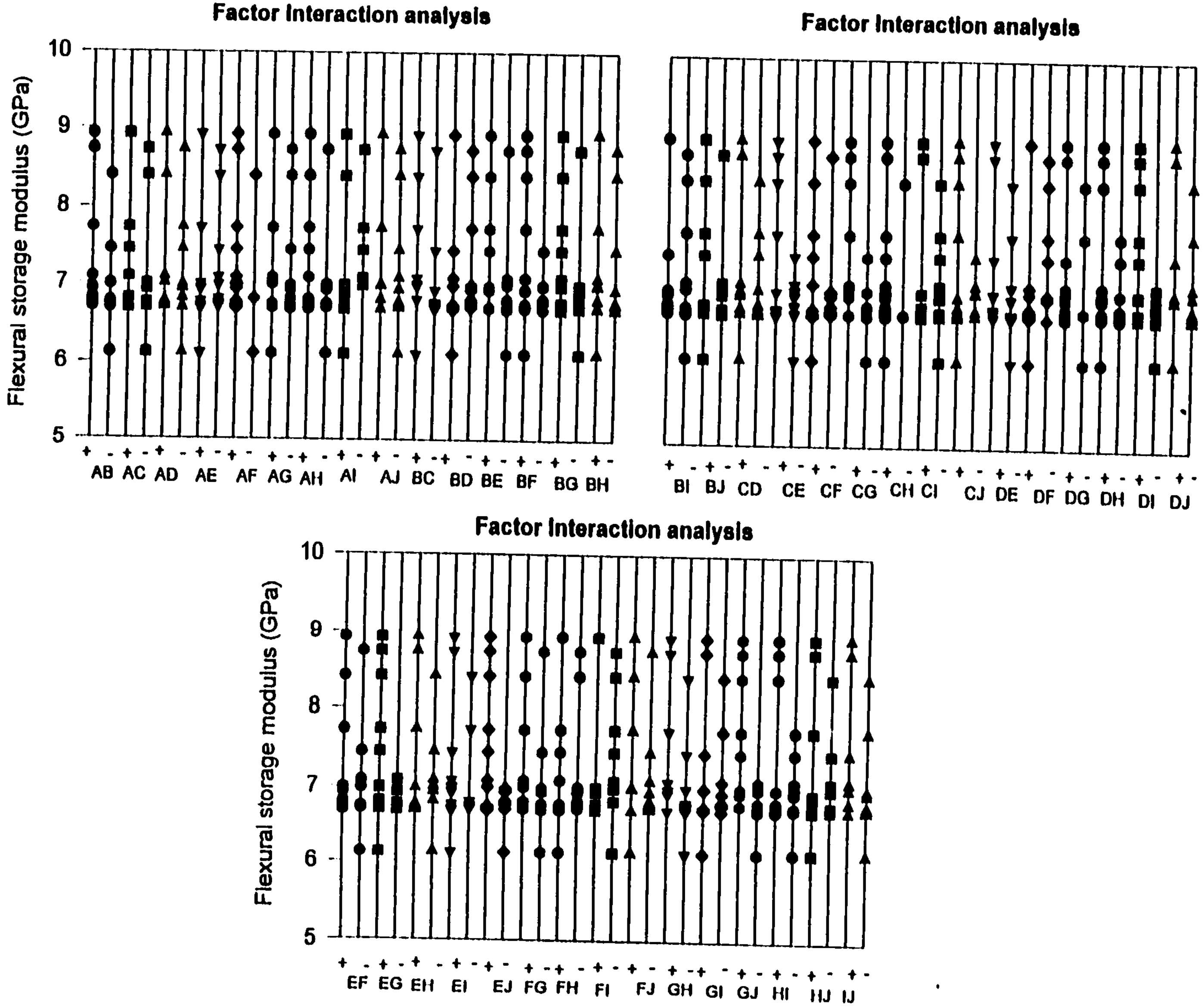


Fig. A.10 - Flexural storage modulus factor interaction analysis.

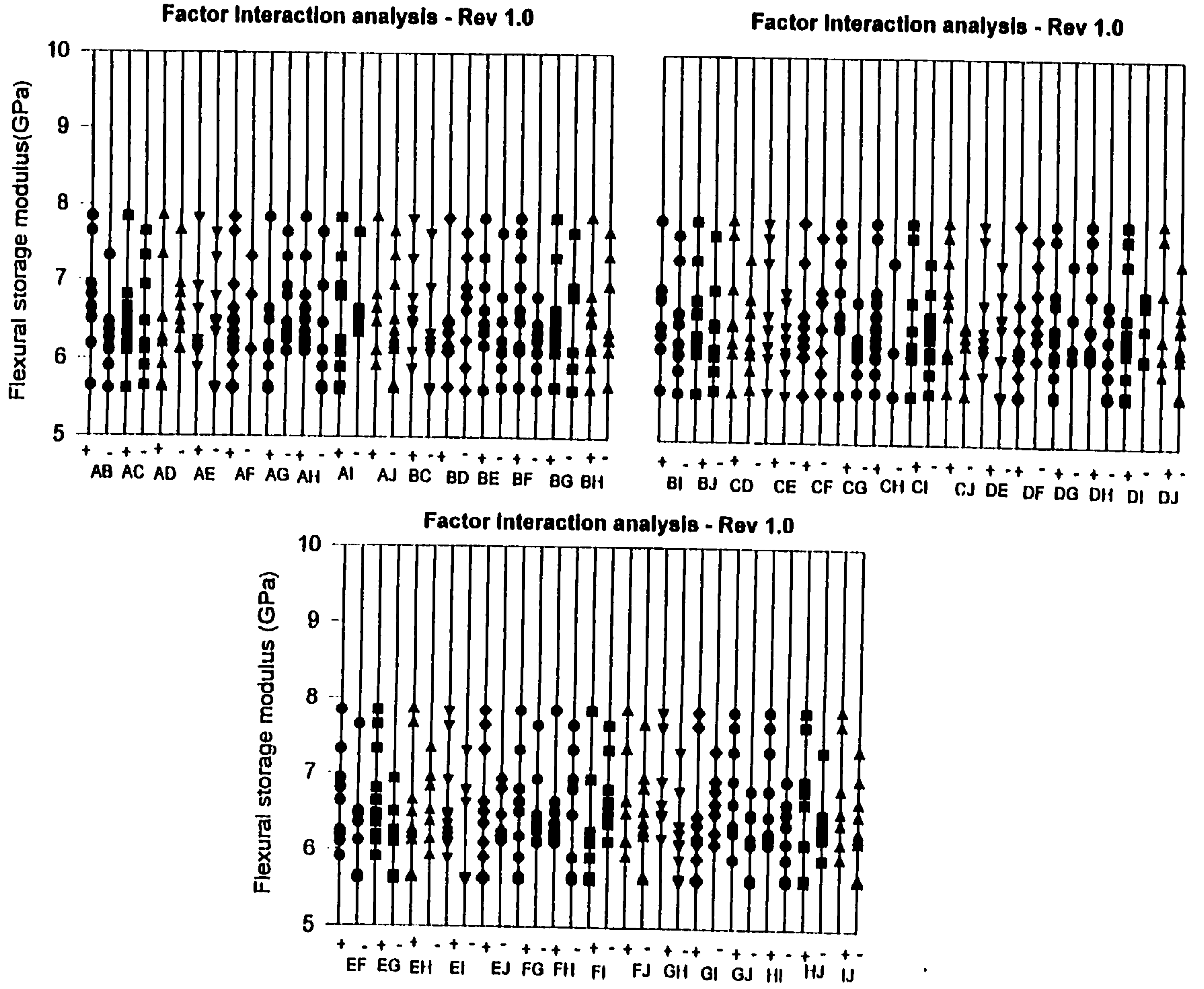


Fig. A.10 - Flexural storage modulus factor interaction analysis.

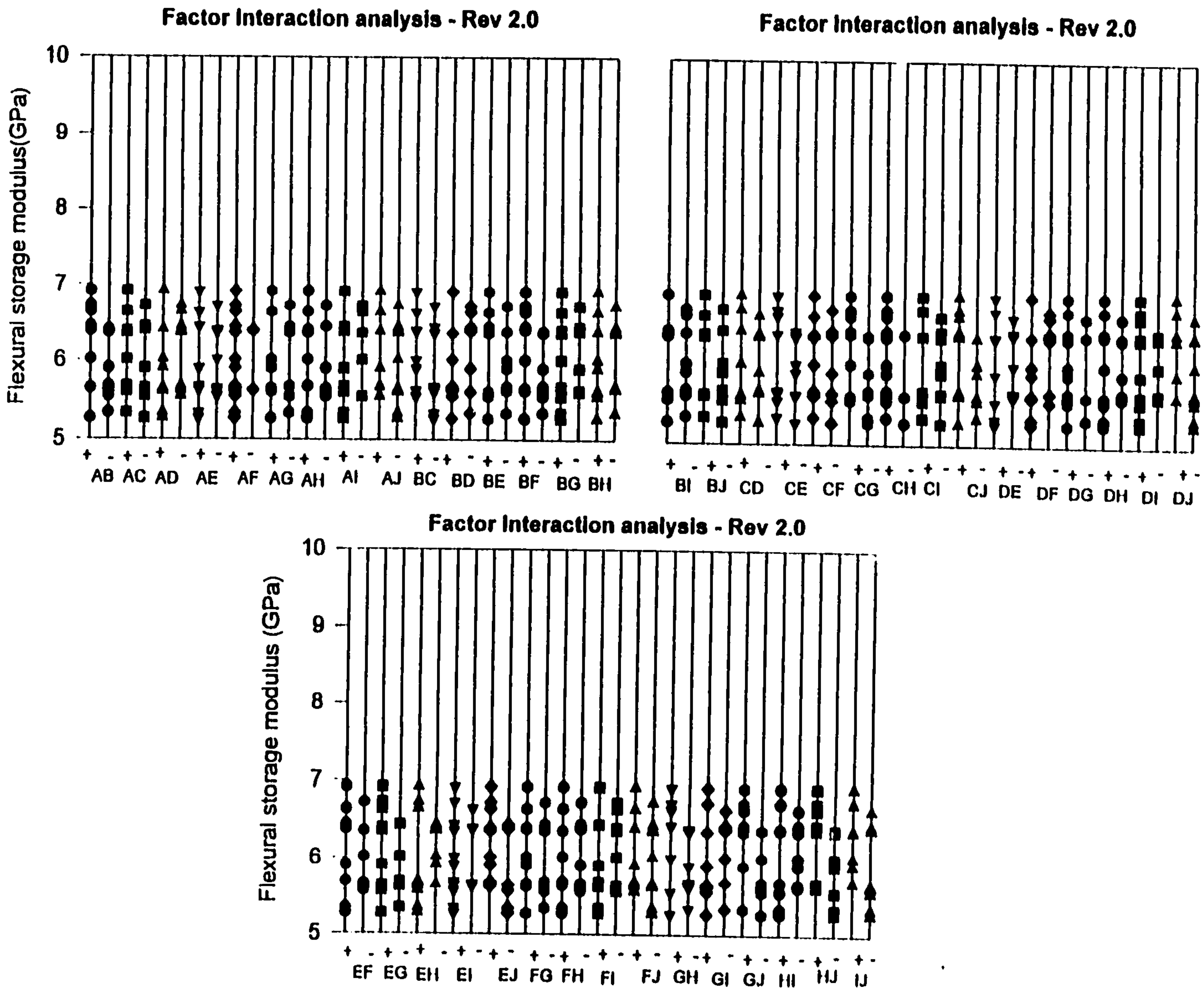


Fig. A.10 - Flexural storage modulus factor interaction analysis.

APPENDIX - B

SEIVE ANALYSIS - CALCULATIONS FOR SCATTER GRAPHS

Table B.1 - Flexural strength (MPa) - Interaction factor sieve analysis.

Run No.	Sieve 0	Sieve 1	Sieve 2	
1	**220.0000**	**191.7983**	**228.3319**	
2	**206.0000**	**174.2067**	**188.1117**	
3	**209.0000**	**149.0050**	**185.5386**	
4	**176.9000**	**176.9000**	**199.5286**	
5	**179.0000**	**179.0000**	**215.5336**	
6	**143.0000**	**143.0000**	**179.5336**	
7	**214.5000**	**186.2983**	**200.2033**	
8	**190.0000**	**190.0000**	**190.0000**	
9	**193.0000**	**193.0000**	**206.9050**	
10	**228.0000**	**196.2067**	**210.1117**	
11	**210.2000**	**178.4067**	**192.3117**	
12	**223.5000**	**191.7067**	**191.7067**	
13	**227.2000**	**167.2050**	**203.7386**	
14	**300.0000**	**240.0050**	**240.0050**	
15	**205.5000**	**177.2983**	**177.2983**	
16	**209.3000**	**149.3050**	**185.8386**	

Table B.2 - Flexural strength (MPa) - Main factor sieve analysis.

Run No.	Sieve 0	Sieve 1	Sieve 2	
1	**219.7820**	**241.1015**	**241.1015**	
2	**205.8710**	**205.8710**	**205.8710**	
3	**208.6100**	**175.4960**	**175.4960**	
4	**176.8730**	**176.8730**	**176.8730**	
5	**178.6360**	**178.6360**	**178.6360**	
6	**142.7460**	**164.0655**	**164.0655**	
7	**214.4640**	**235.7835**	**215.7376**	
8	**189.9330**	**211.2525**	**191.2066**	
9	**192.6840**	**214.0035**	**214.0035**	
10	**227.8170**	**194.7030**	**174.6571**	
11	**210.2030**	**210.2030**	**210.2030**	
12	**223.5240**	**223.5240**	**203.4781**	
13	**227.1960**	**194.0820**	**174.0361**	
14	**299.2560**	**266.1420**	**246.0961**	
15	**205.4090**	**205.4090**	**205.4090**	
16	**209.2970**	**197.5025**	**177.4566**	

Table B.3 - Flexural modulus (GPa) - Interaction factor sieve analysis.

Run No.	Sieve 0	Sieve 1	Sieve 2	
1	**6.9854**	**6.4733**	**5.5551**	
2	**6.7694**	**6.2573**	**5.3391**	
3	**6.6992**	**6.1871**	**5.2689**	
4	**7.7316**	**6.6392**	**6.6392**	
5	**7.0880**	**6.5077**	**6.0159**	
6	**8.7336**	**7.6412**	**6.7230**	
7	**7.4504**	**6.3580**	**6.3580**	
8	**8.4081**	**7.3157**	**6.3975**	
9	**6.1216**	**6.1216**	**5.6298**	
10	**6.9982**	**5.9058**	**5.9058**	
11	**6.6845**	**6.1042**	**5.6778**	
12	**6.7075**	**5.6151**	**5.6151**	
13	**6.9328**	**6.9328**	**6.4410**	
14	**6.7428**	**5.6504**	**5.6504**	
15	**6.8064**	**6.8064**	**6.3800**	
16	**8.9292**	**7.8368**	**6.9186**	

Table B.4 - Flexural modulus (GPa) - Main factor sieve analysis.

Run No.	Sieve 0	Sieve 1	Sieve 2	
1	**6.9854**	**5.9760**	**5.9760**	
2	**6.7694**	**6.0508**	**6.7219**	
3	**6.6992**	**6.6992**	**6.6992**	
4	**7.7316**	**7.7316**	**6.8467**	
5	**7.0880**	**7.0880**	**6.2031**	
6	**8.7336**	**7.0056**	**7.0056**	
7	**7.4504**	**6.4410**	**6.4410**	
8	**8.4081**	**8.4081**	**7.5232**	
9	**6.1216**	**6.1216**	**5.9078**	
10	**6.9982**	**6.2796**	**6.2796**	
11	**6.6845**	**5.9659**	**6.6370**	
12	**6.7075**	**5.9889**	**5.9889**	
13	**6.9328**	**6.9328**	**7.6039**	
14	**6.7428**	**6.7428**	**6.7428**	
15	**6.8064**	**5.7970**	**6.4681**	
16	**8.9292**	**7.2012**	**6.9874**	

Table B.5 - Shear strength (MPa) - Interaction factor sieve analysis.

Run No.	Sieve 0	Sieve 1	Sieve 2	Sieve 3
1	**27.1011**	**23.8780**	**28.2733**	**28.2733**
2	**23.5817**	**23.5817**	**27.9770**	**27.9770**
3	**24.7531**	**21.5300**	**25.9253**	**27.3664**
4	**26.0828**	**26.0828**	**26.0828**	**27.5239**
5	**15.9709**	**15.9709**	**20.3662**	**24.4002**
6	**18.6947**	**18.6947**	**23.0900**	**25.6829**
7	**25.8952**	**22.6721**	**22.6721**	**25.2650**
8	**19.7791**	**19.7791**	**24.1744**	**25.6155**
9	**18.0572**	**18.0572**	**22.4525**	**22.4525**
10	**25.2708**	**25.2708**	**25.2708**	**29.3048**
11	**25.5817**	**25.5817**	**25.5817**	**28.1746**
12	**25.8664**	**25.8664**	**25.8664**	**27.3075**
13	**27.4262**	**24.2031**	**28.5984**	**28.5984**
14	**25.8397**	**22.6166**	**22.6166**	**24.0577**
15	**26.6924**	**23.4693**	**23.4693**	**27.5033**
16	**20.8865**	**17.6634**	**22.0587**	**26.0927**

Table B.6 - Shear strength (MPa) - Main factor sieve analysis.

Run No.	Sieve 0	Sieve 1	Sieve 2	
1	**24.1011**	**24.1011**	**26.0896**	
2	**23.5817**	**23.5817**	**23.5817**	
3	**24.7531**	**24.7531**	**24.7531**	
4	**26.0828**	**30.8781**	**28.1078**	
5	**15.9709**	**20.7662**	**20.7662**	
6	**18.6947**	**18.6947**	**20.6832**	
7	**25.8952**	**25.8952**	**27.8837**	
8	**19.7791**	**24.5744**	**23.7926**	
9	**18.0572**	**22.8525**	**24.8510**	
10	**25.2708**	**25.2708**	**25.2708**	
11	**25.5817**	**25.5817**	**22.8114**	
12	**25.8664**	**25.8664**	**25.8664**	
13	**27.4262**	**27.4262**	**24.6559**	
14	**25.8397**	**25.8397**	**25.8397**	
15	**26.6924**	**26.6924**	**23.9221**	
16	**20.8865**	**25.6818**	**24.9000**	

Table B.7 - Damping ratio - Interaction factor sieve analysis.

Run No.	Sieve 0	Sieve 1	Sieve 2	
1	0.2225	0.2485	0.2970	
2	0.2024	0.2963	0.2963	
3	0.2239	0.3178	0.3178	
4	0.2312	0.2572	0.3057	
5	0.2063	0.3002	0.3002	
6	0.1680	0.2619	0.2619	
7	0.2090	0.3029	0.3029	
8	0.3965	0.3286	0.3286	
9	0.3320	0.2641	0.2641	
10	0.1987	0.2247	0.2732	
11	0.2442	0.2702	0.3187	
12	0.1905	0.2844	0.2844	
13	0.2064	0.3003	0.3003	
14	0.2143	0.3082	0.3082	
15	0.2506	0.2506	0.2991	
16	0.2261	0.2521	0.3006	

Table B.8 - Damping ratio - Main factor sieve analysis.

Run No.	Sieve 0	Sieve 1	sieve 2	
1	0.2225	0.2225	0.2567	
2	0.2024	0.2453	0.2795	
3	0.2239	0.2239	0.2581	
4	0.2312	0.1833	0.1833	
5	0.2063	0.1584	0.1926	
6	0.1680	0.2109	0.2109	
7	0.2090	0.2090	0.2090	
8	0.3965	0.3486	0.3486	
9	0.3320	0.2841	0.2841	
10	0.1987	0.2416	0.2416	
11	0.2442	0.2871	0.2871	
12	0.1905	0.2334	0.2676	
13	0.2064	0.2064	0.2406	
14	0.2143	0.2143	0.2485	
15	0.2506	0.2506	0.2506	
16	0.2261	0.2211	0.2553	

Table B.9 - Glass transition temperature (° C) - Interaction factor sieve analysis.

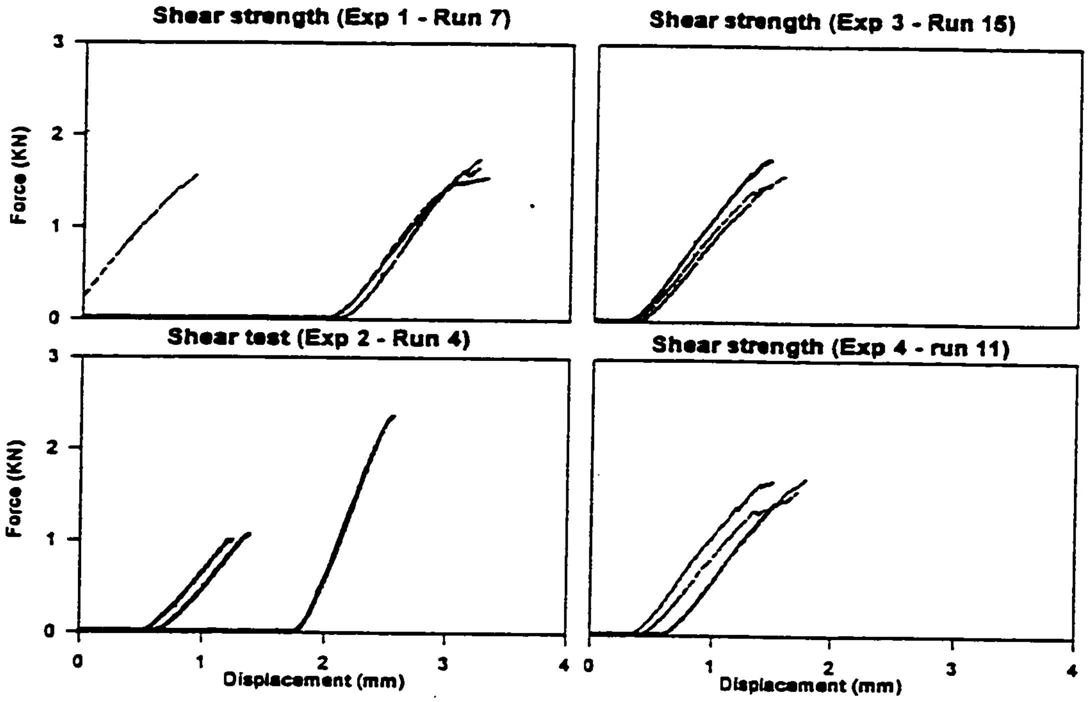
Run No.	Sieve 0	Sieve 1	Sieve 2	
1	**109.2180**	**96.9966**	**83.9896**	
2	**94.2366**	**78.1819**	**78.1819**	
3	**114.1490**	**98.0943**	**73.7074**	
4	**140.4790**	**112.2119**	**100.8320**	
5	**91.1392**	**78.9178**	**78.9178**	
6	**83.2516**	**83.2516**	**71.8717**	
7	**124.7860**	**112.5646**	**99.5576**	
8	**141.9290**	**113.6529**	**102.2730**	
9	**109.8440**	**97.6226**	**86.2427**	
10	**102.2870**	**90.0656**	**90.0656**	
11	**137.3360**	**109.0599**	**97.6800**	
12	**107.6630**	**107.6630**	**107.6630**	
13	**128.2980**	**128.2980**	**103.9111**	
14	**120.1790**	**120.1790**	**95.7921**	
15	**130.4370**	**114.3823**	**89.9954**	
16	**132.7520**	**104.4849**	**80.0980**	

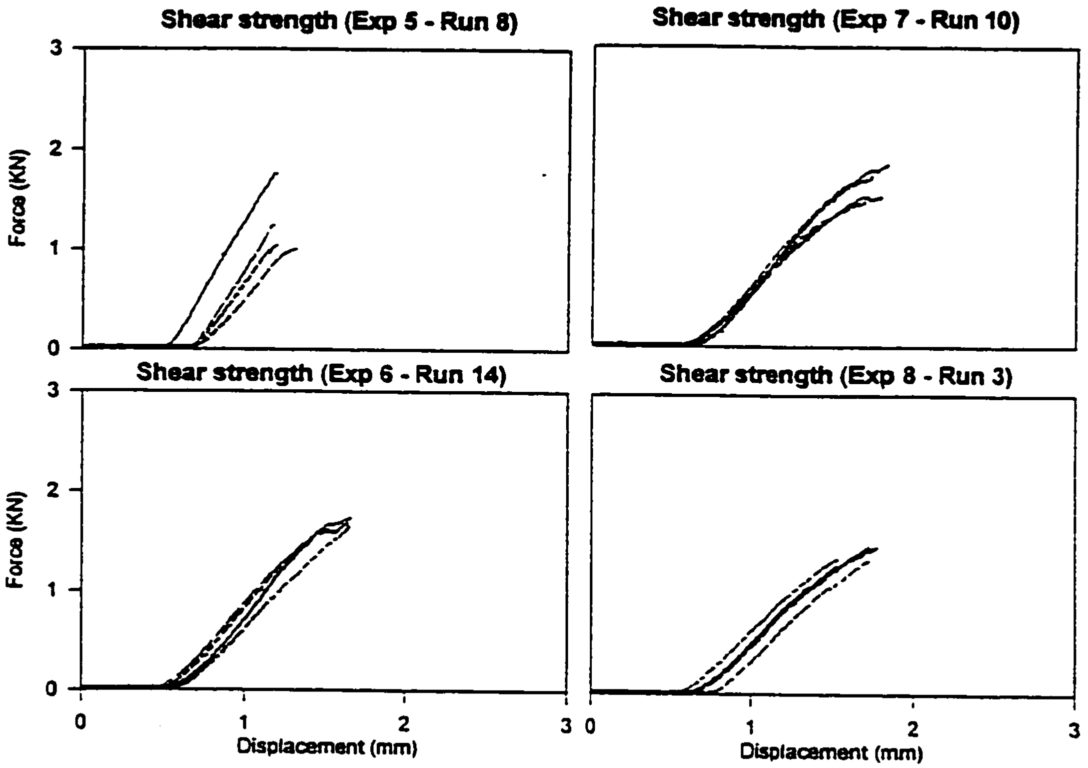
Table B.10 - Glass transition temperature (° C) - Main factor sieve analysis.

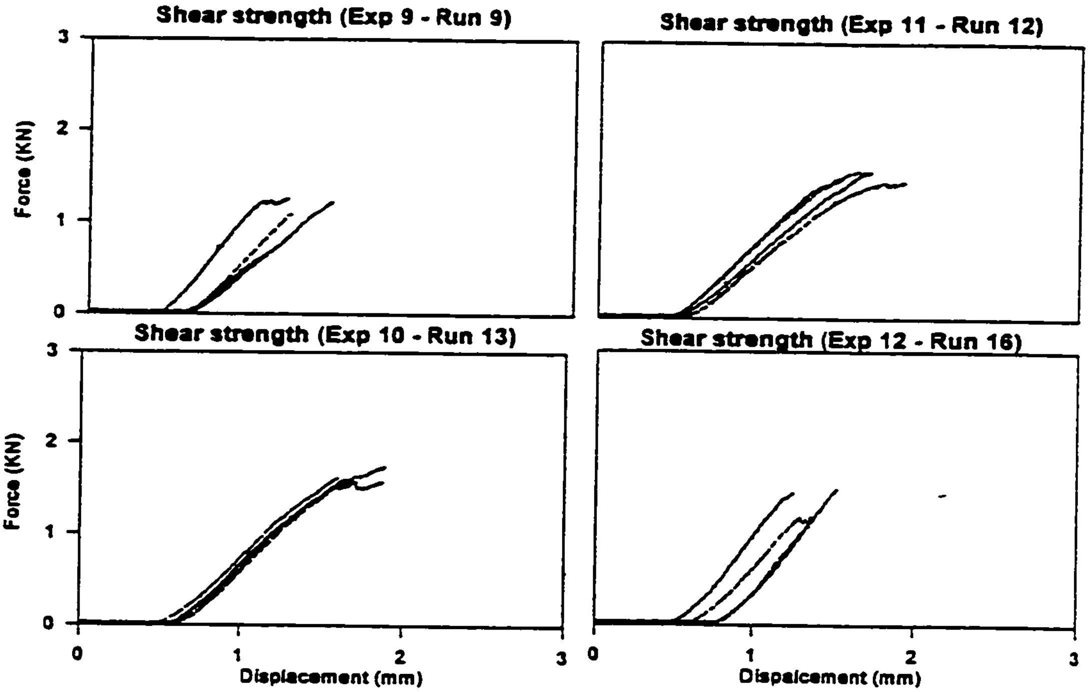
Run No.	Sieve 0	Sieve 1	Sieve 2	
1	**109.2180**	**109.2180**	**109.2180**	
2	**94.2366**	**94.2366**	**103.8712**	
3	**114.1487**	**114.1487**	**107.5523**	
4	**140.4787**	**112.2912**	**112.2912**	
5	**91.1392**	**91.1392**	**91.1392**	
6	**83.2516**	**83.2516**	**92.8862**	
7	**124.7857**	**124.7857**	**118.1893**	
8	**141.9287**	**113.7412**	**113.7412**	
9	**109.8443**	**109.8443**	**109.8443**	
10	**102.2870**	**102.2870**	**105.3252**	
11	**137.3360**	**109.1485**	**118.7831**	
12	**107.6633**	**107.6633**	**117.2979**	
13	**128.2977**	**100.1102**	**93.5138**	
14	**120.1793**	**120.1793**	**113.5829**	
15	**130.4373**	**102.2498**	**102.2498**	
16	**132.7520**	**104.4645**	**107.5027**	

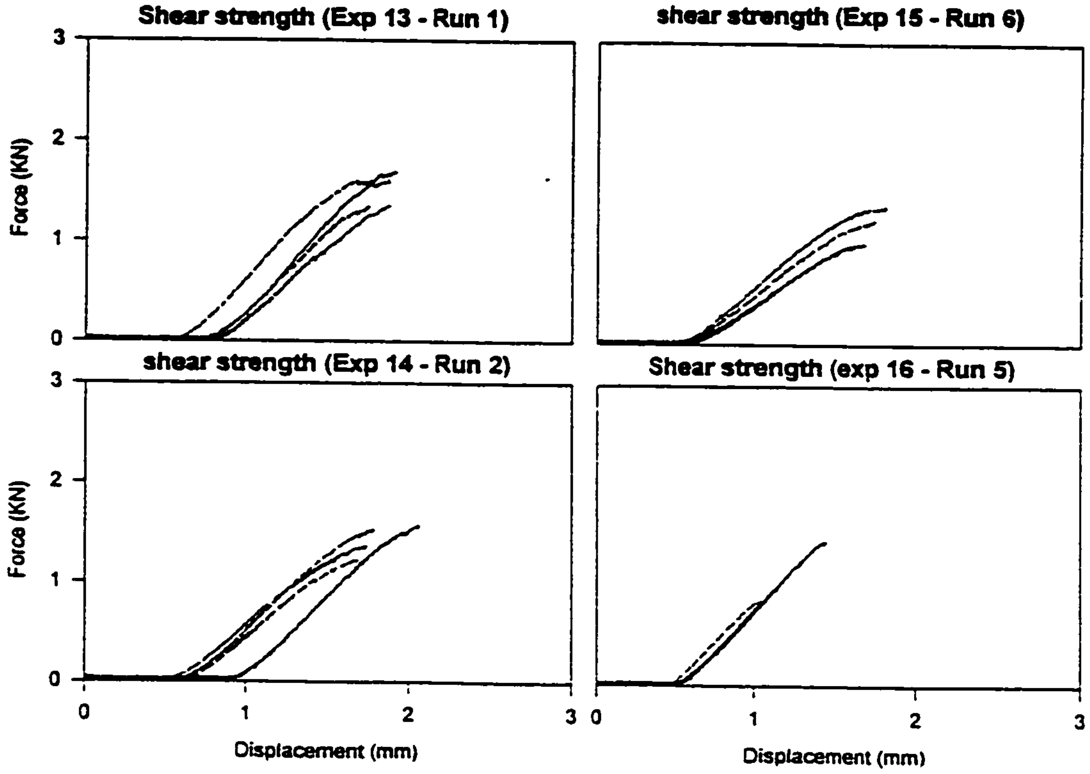
APPENDIX - C

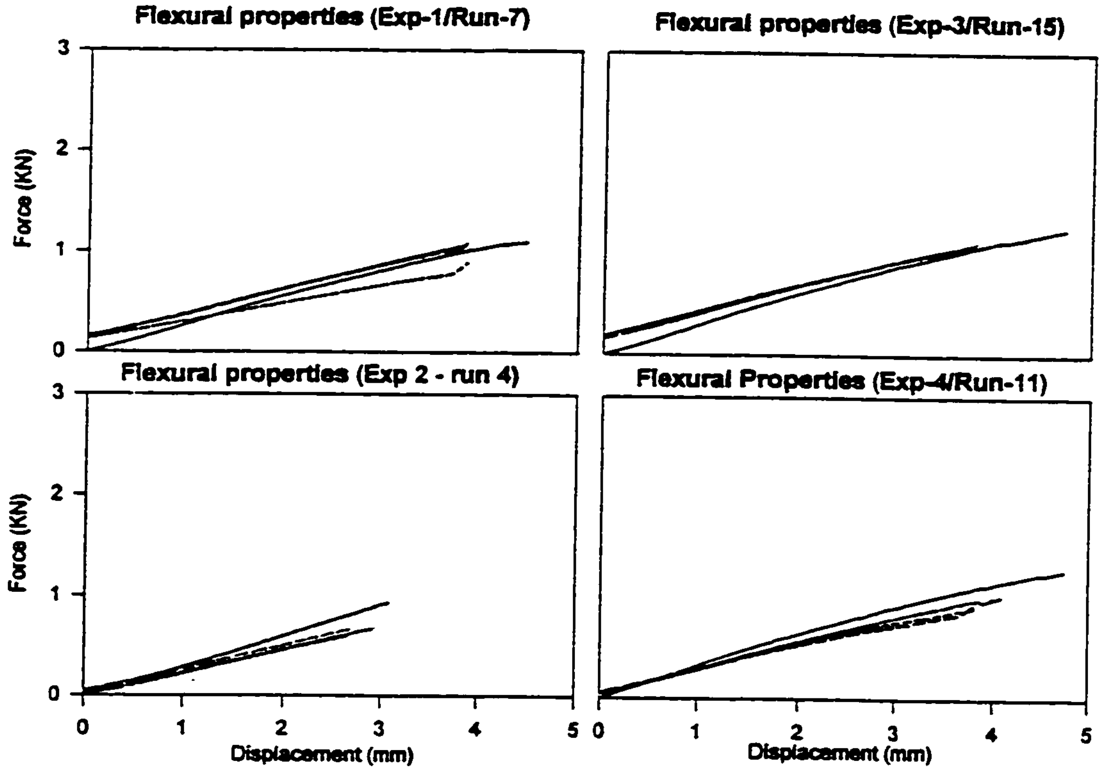
SEIVE ANALYSIS - GRAPHS OF ALL MECHANICAL TESTS

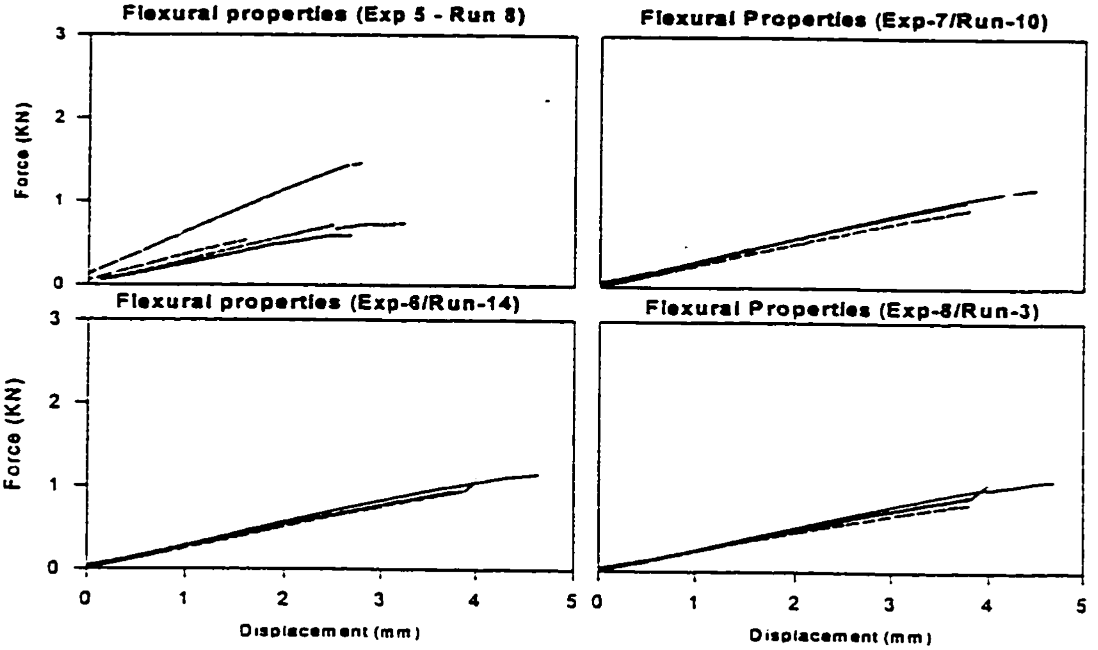

Shear strength (Exp 1 - Run 7)
Shear strength (Exp 3 - Run 15)
Shear test (Exp 2 - Run 4)
Shear strength (Exp 4 - run 11)
Force (KN)
Force (KN)
0
1
2
3
0
1
2
3
4
Displacement (mm)
Displacement (mm)

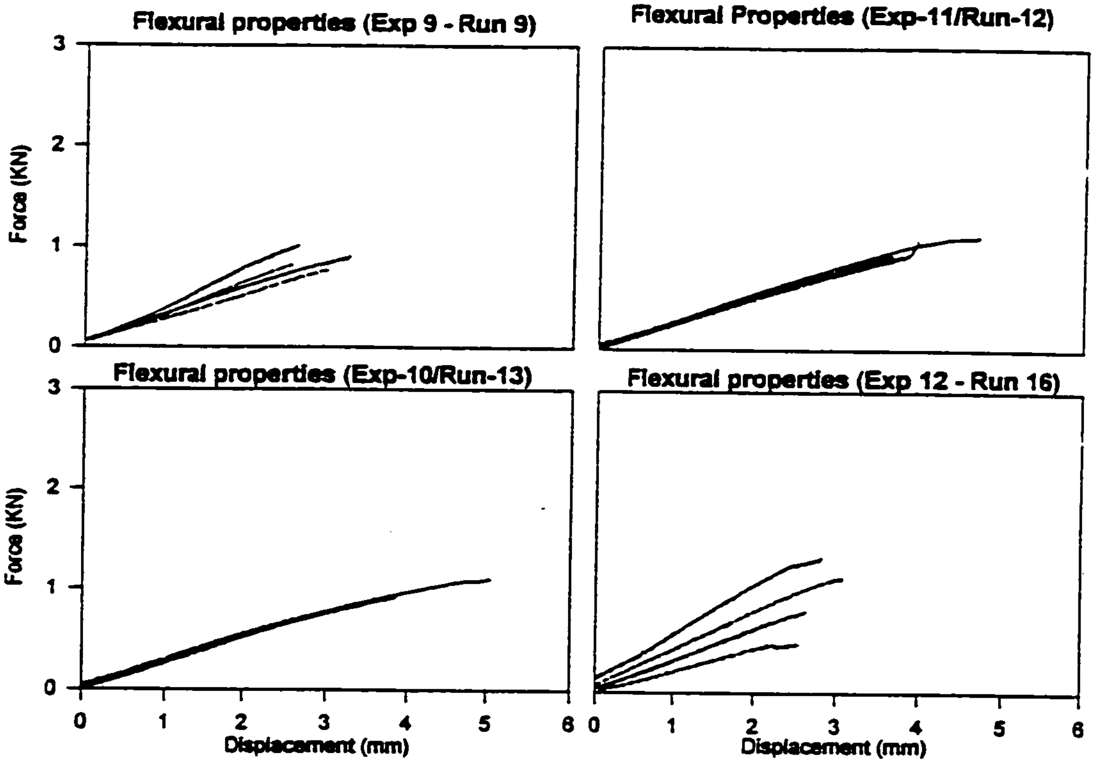

Shear strength (Exp 5 - Run 8)
Shear strength (Exp 7 - Run 10)
Shear strength (Exp 6 - Run 14)
Shear strength (Exp 8 - Run 3)
Force (KN)
Force (KN)
0
1
2
3
Displacement (mm)
Displacement (mm)


Shear strength (Exp 9 - Run 9)
Shear strength (Exp 11 - Run 12)
Shear strength (Exp 10 - Run 13)
Shear strength (Exp 12 - Run 16)
Force (KN)
Force (KN)
0
1
2
3
Displacement (mm)
Dispalcement (mm)


Shear strength (Exp 13 - Run 1)
shear strength (Exp 15 - Run 6)
shear strength (Exp 14 - Run 2)
Shear strength (exp 16 - Run 5)
Force (KN)
Force (KN)
0
1
2
3
Displacement (mm)
Displacement (mm)


Flexural properties (Exp-1/Run-7)
Flexural properties (Exp-3/Run-15)
Flexural properties (Exp 2 - run 4)
Flexural Properties (Exp-4/Run-11)
Force (KN)
Force (KN)
0
1
2
3
0
1
2
3
4
5
Displacement (mm)
Displacement (mm)


Flexural properties (Exp 5 - Run 8)
Flexural Properties (Exp-7/Run-10)
Flexural properties (Exp-6/Run-14)
Flexural Properties (Exp-8/Run-3)
Force (KN)
Force (KN)
0
1
2
3
0
1
2
3
4
5
Displacement (mm)
Displacement (mm)

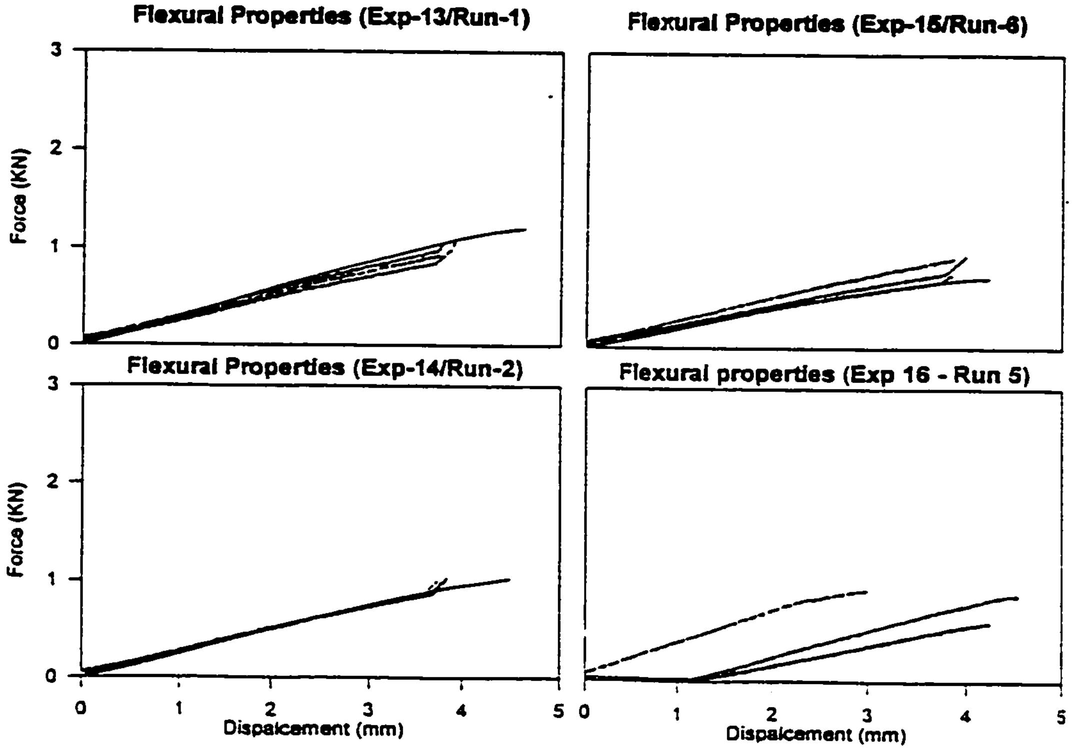


Flexural properties (Exp 9 - Run 9)

Flexural Properties (Exp-11/Run-12)

Flexural properties (Exp-10/Run-13)

Flexural properties (Exp 12 - Run 16)

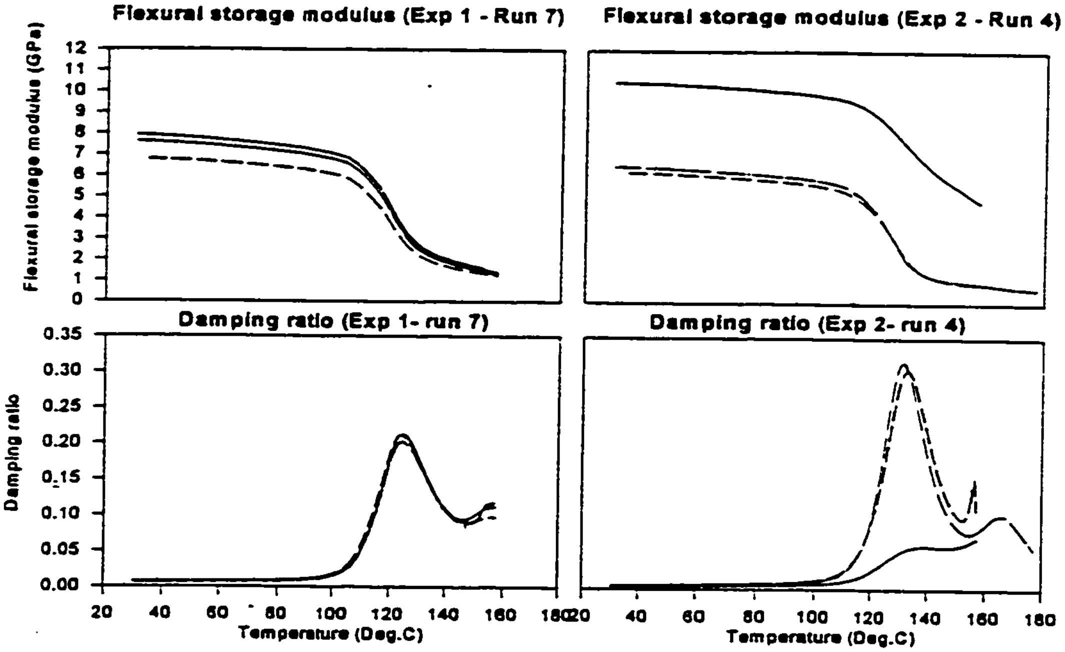


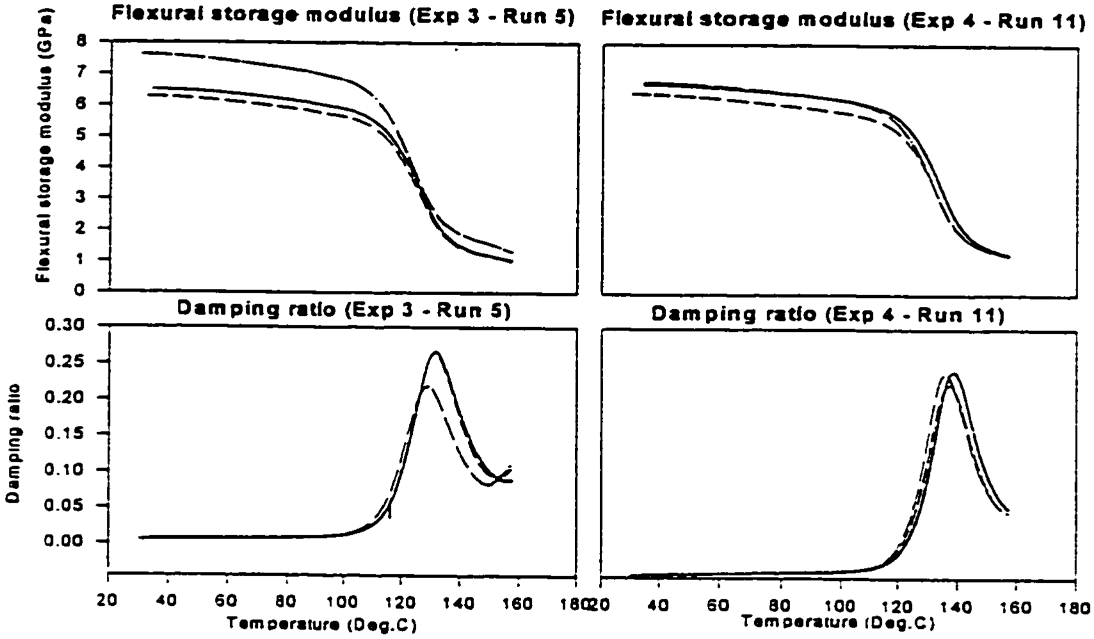
Flexural Properties (Exp-13/Run-1)

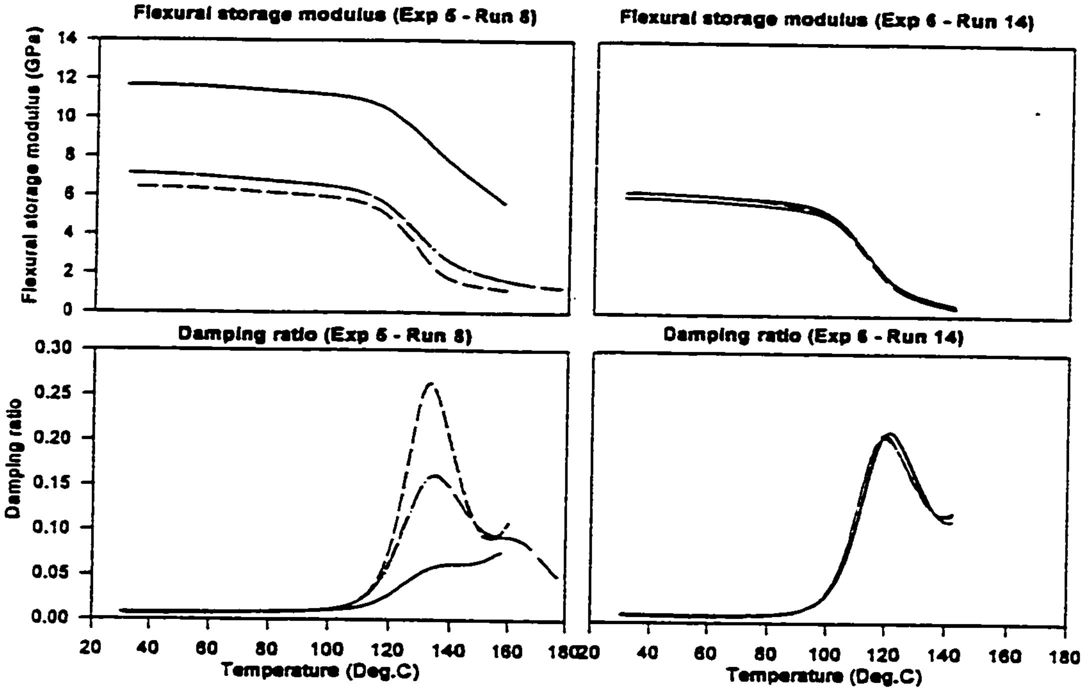
Flexural Properties (Exp-15/Run-6)

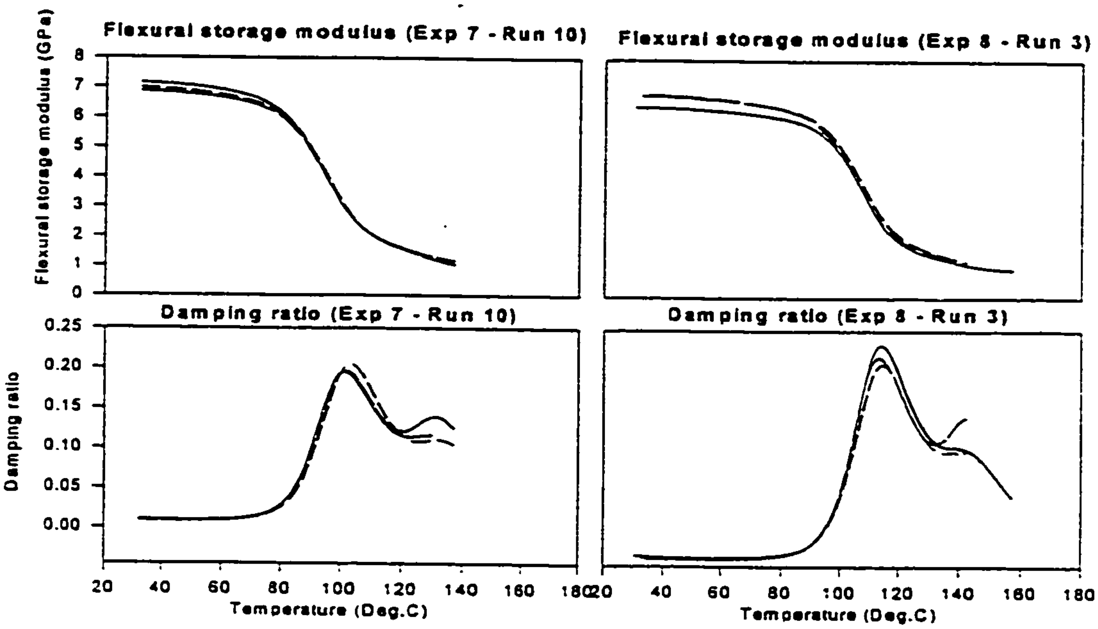
Flexural Properties (Exp-14/Run-2)

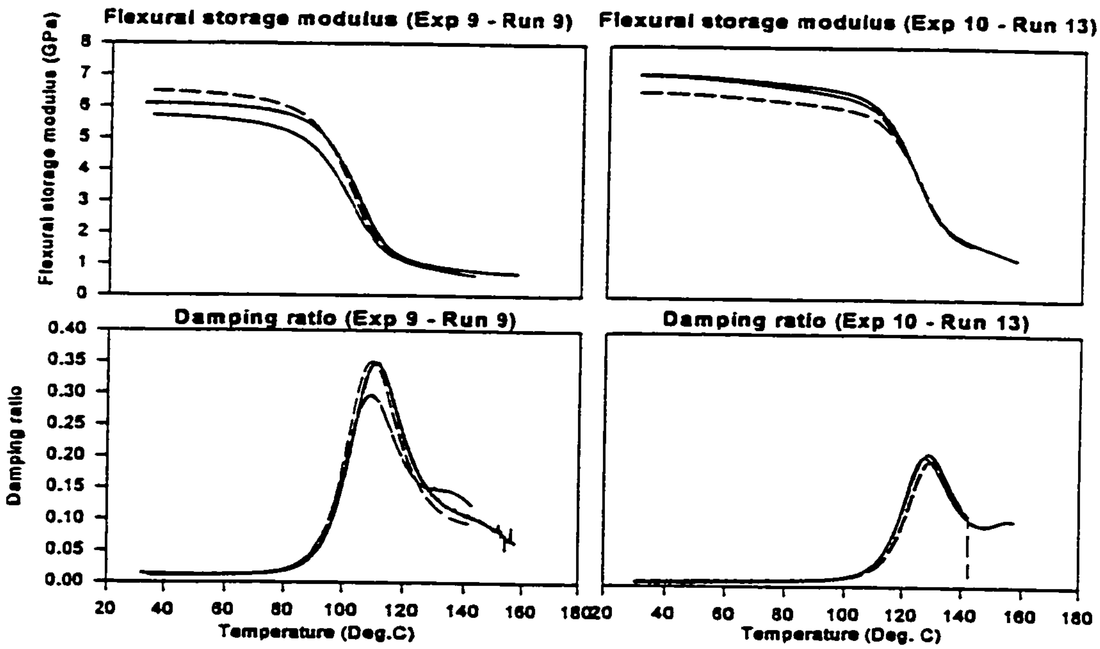
Flexural properties (Exp 16 - Run 5)

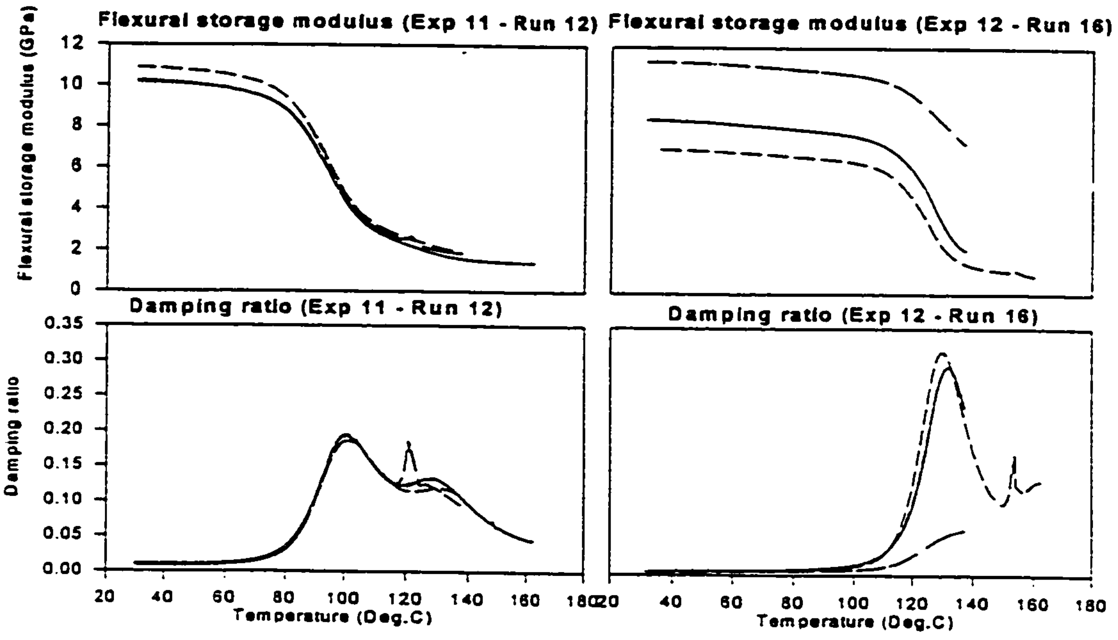

Flexural storage modulus (Exp 1 - Run 7)
Flexural storage modulus (Exp 2 - Run 4)
Flexural storage modulus (GPa)
12
11
10
9
8
7
6
5
4
3
2
1
0
Damping ratio (Exp 1- run 7)
Damping ratio (Exp 2- run 4)
Damping ratio
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
20
40
60
80
100
120
140
160
180
Temperature (Deg.C)
Temperature (Deg.C)

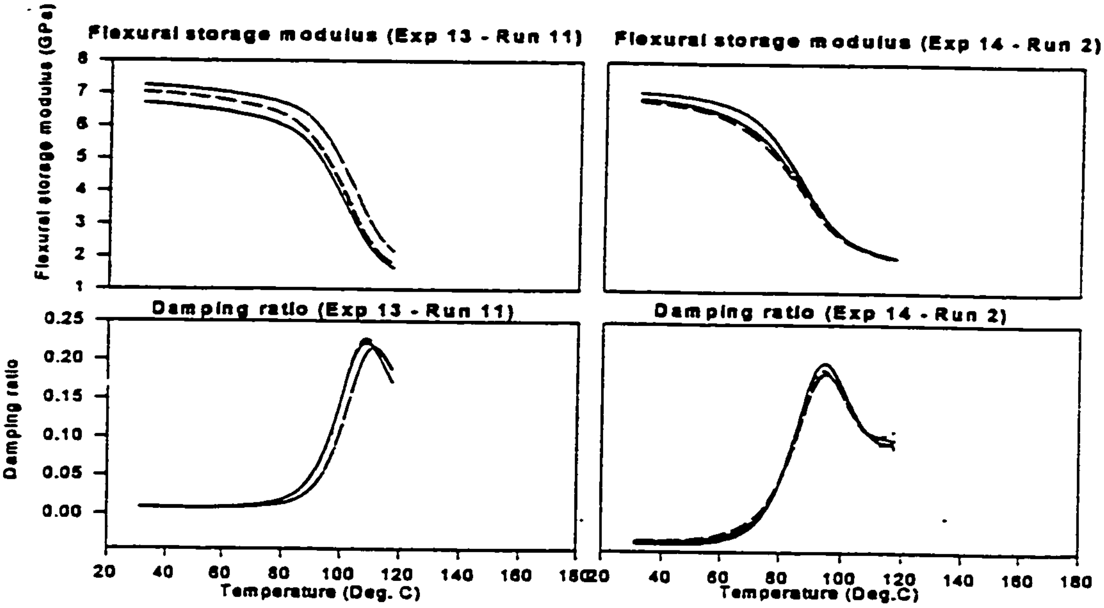

Flexural storage modulus (Exp 3 - Run 5)
Flexural storage modulus (Exp 4 - Run 11)
Flexural storage modulus (GPa)
8
7
6
5
4
3
2
1
0
Damping ratio (Exp 3 - Run 5)
Damping ratio (Exp 4 - Run 11)
Damping ratio
0.30
0.25
0.20
0.15
0.10
0.05
0.00
20
40
60
80
100
120
140
160
180
Temperature (Deg.C)
Temperature (Deg.C)


Flexural storage modulus (Exp 5 - Run 8)
Flexural storage modulus (Exp 6 - Run 14)
Flexural storage modulus (GPa)
14
12
10
8
6
4
2
0
Damping ratio (Exp 5 - Run 8)
Damping ratio (Exp 6 - Run 14)
Damping ratio
0.30
0.25
0.20
0.15
0.10
0.05
0.00
20
40
60
80
100
120
140
160
180
Temperature (Deg.C)
Temperature (Deg.C)


Flexural storage modulus (Exp 7 - Run 10)
Flexural storage modulus (Exp 8 - Run 3)
Flexural storage modulus (GPa)
8
7
6
5
4
3
2
1
0
Damping ratio (Exp 7 - Run 10)
Damping ratio (Exp 8 - Run 3)
Damping ratio
0.25
0.20
0.15
0.10
0.05
0.00
20
40
60
80
100
120
140
160
180
Temperature (Deg.C)
Temperature (Deg.C)


Flexural storage modulus (Exp 9 - Run 9)
Flexural storage modulus (Exp 10 - Run 13)
Flexural storage modulus (GPa)
0
1
2
3
4
5
6
7
8
Damping ratio (Exp 9 - Run 9)
Damping ratio (Exp 10 - Run 13)
Damping ratio
0.00
0.05
0.10
0.15
0.20
0.25
0.30
0.35
0.40
20
40
60
80
100
120
140
160
180
Temperature (Deg.C)
Temperature (Deg. C)

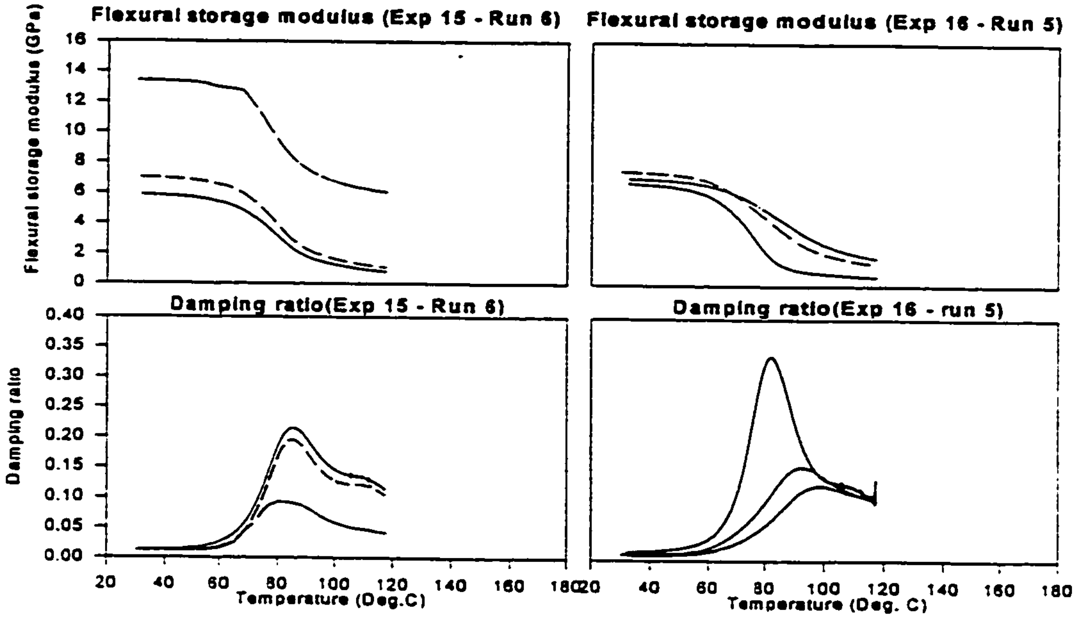

Flexural storage modulus (Exp 11 - Run 12)
Flexural storage modulus (Exp 12 - Run 16)
Flexural storage modulus (GPa)
0
2
4
6
8
10
12
Damping ratio (Exp 11 - Run 12)
Damping ratio (Exp 12 - Run 16)
Damping ratio
0.00
0.05
0.10
0.15
0.20
0.25
0.30
0.35
20
40
60
80
100
120
140
160
180
Temperature (Deg.C)
Temperature (Deg.C)


Flexural storage modulus (Exp 13 - Run 11)

Flexural storage modulus (Exp 14 - Run 2)

Damping ratio (Exp 13 - Run 11)

Damping ratio (Exp 14 - Run 2)


Flexural storage modulus (Exp 15 - Run 6)

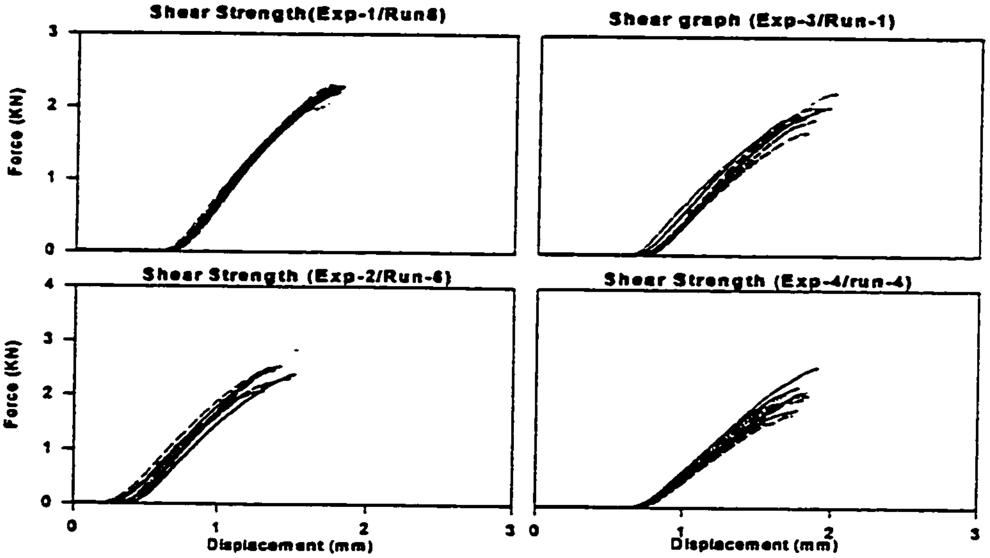
Flexural storage modulus (Exp 16 - Run 5)

Damping ratio(Exp 15 - Run 6)

Damping ratio(Exp 16 - run 5)

APPENDIX - D

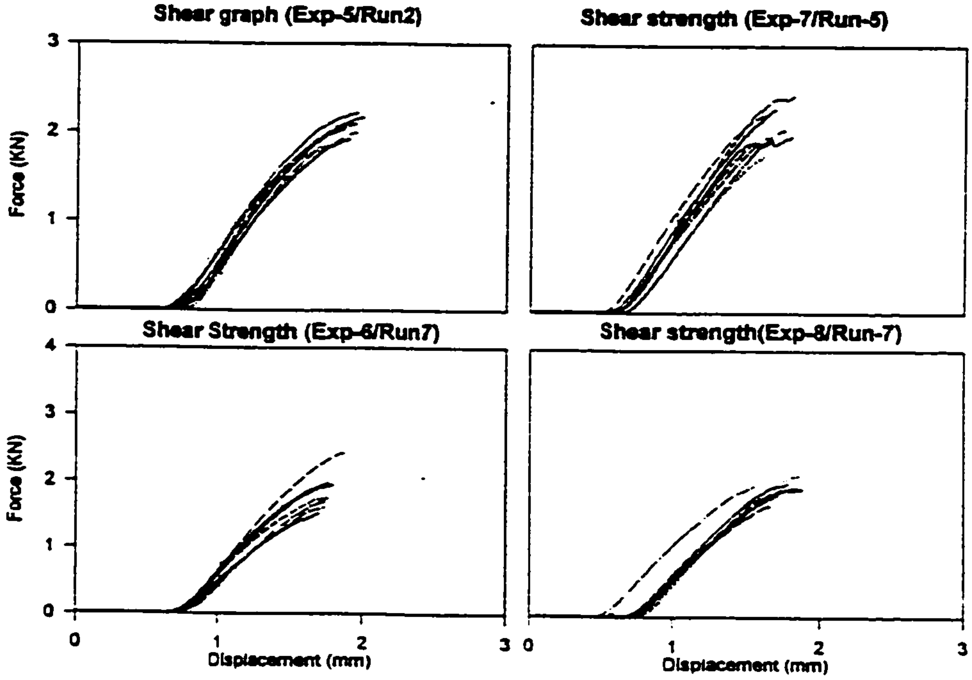
DESIGN OF EXPERIMENTS - GRAPHS OF ALL MECHANICAL TESTS


Shear Strength(Exp-1/Run8)

Shear graph (Exp-3/Run-1)

Shear Strength (Exp-2/Run-6)

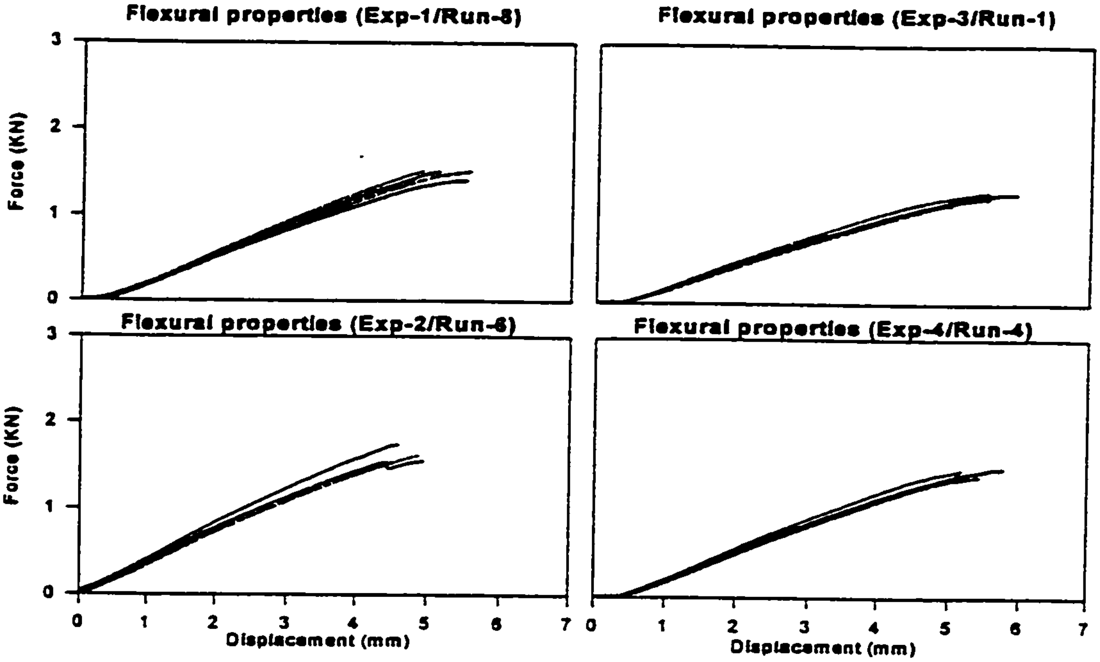
Shear Strength (Exp-4/run-4)


Shear graph (Exp-5/Run2)

Shear strength (Exp-7/Run-5)

Shear Strength (Exp-6/Run7)

Shear strength(Exp-8/Run-7)

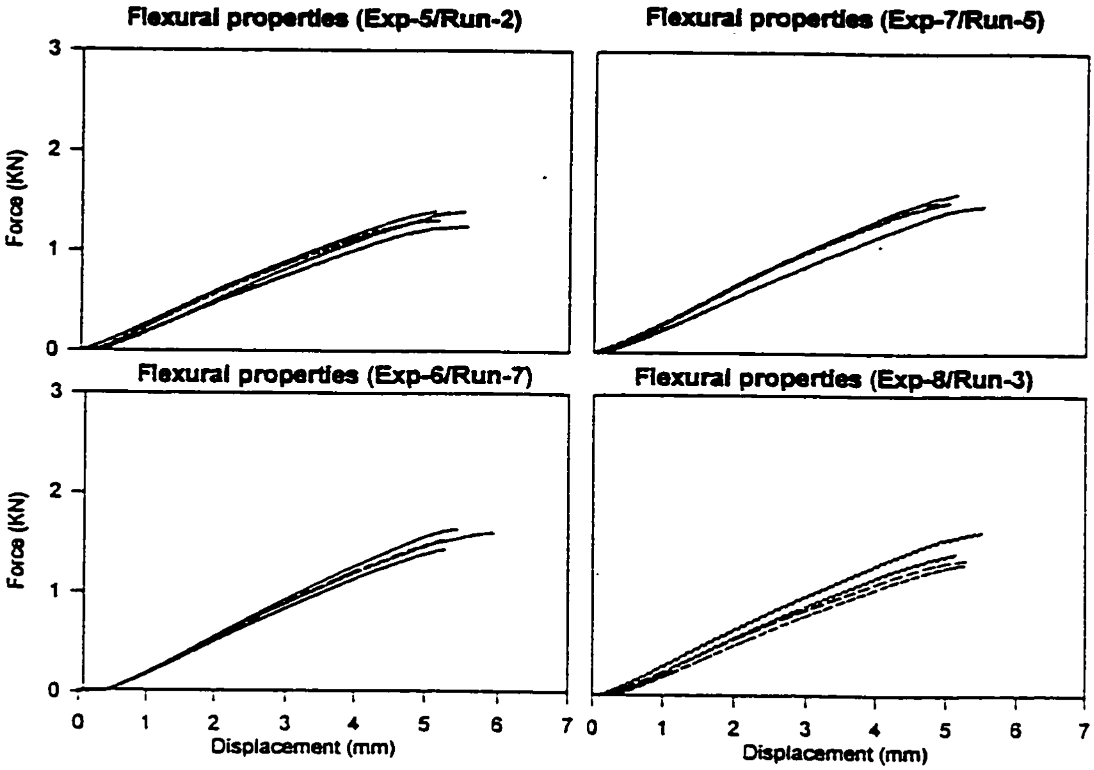


Flexural properties (Exp-1/Run-8)

Flexural properties (Exp-3/Run-1)

Flexural properties (Exp-2/Run-6)

Flexural properties (Exp-4/Run-4)

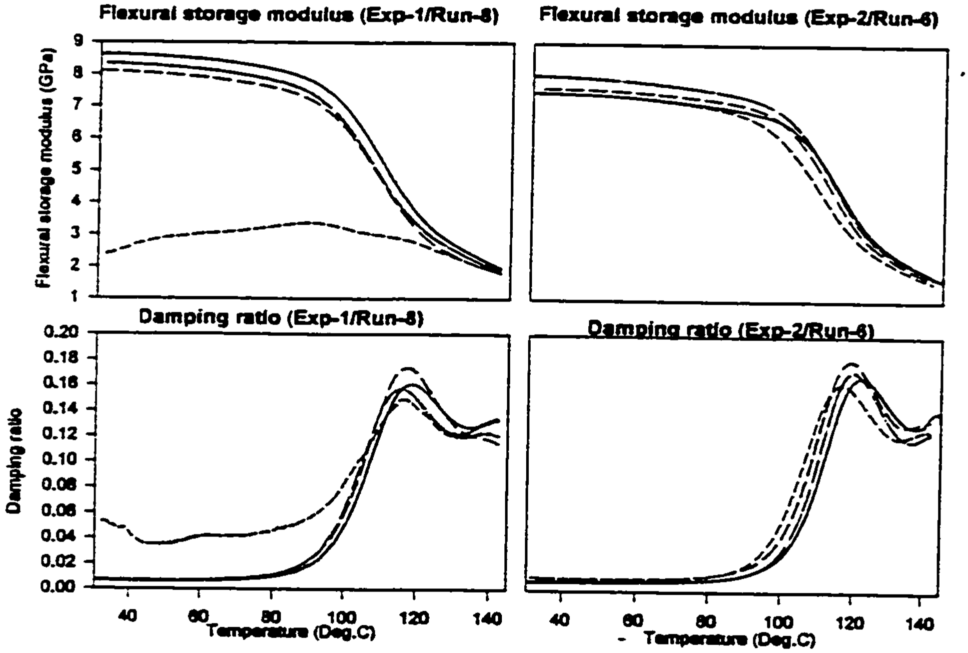


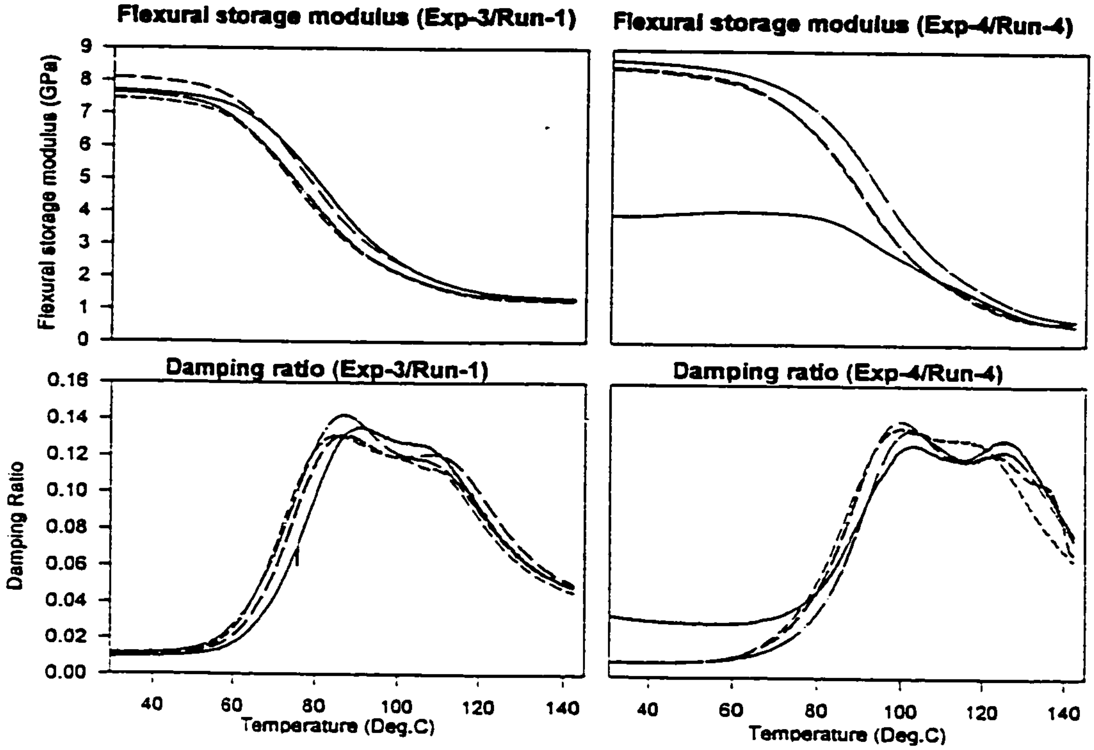
Flexural properties (Exp-5/Run-2)

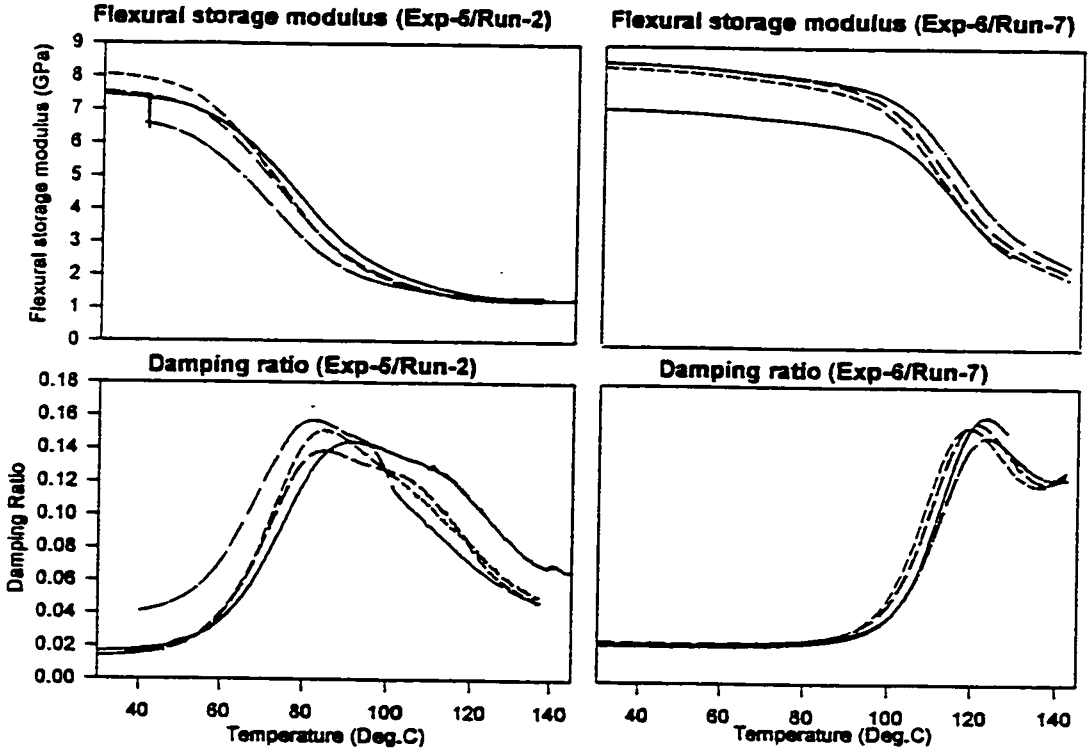
Flexural properties (Exp-7/Run-5)

Flexural properties (Exp-6/Run-7)

Flexural properties (Exp-8/Run-3)


Flexural storage modulus (Exp-1/Run-8)
Flexural storage modulus (Exp-2/Run-6)
Flexural storage modulus (GPa)
9
8
7
6
5
4
3
2
1
Damping ratio (Exp-1/Run-8)
Damping ratio (Exp-2/Run-6)
Damping ratio
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
40
60
80
100
120
140
Temperature (Deg.C)


Flexural storage modulus (Exp-3/Run-1)
Flexural storage modulus (Exp-4/Run-4)
Flexural storage modulus (GPa)
9
8
7
6
5
4
3
2
1
0
Damping ratio (Exp-3/Run-1)
Damping ratio (Exp-4/Run-4)
Damping Ratio
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
40
60
80
100
120
140
Temperature (Deg.C)


Flexural storage modulus (Exp-5/Run-2)
Flexural storage modulus (Exp-6/Run-7)
Flexural storage modulus (GPa)
0
1
2
3
4
5
6
7
8
9
Damping ratio (Exp-5/Run-2)
Damping ratio (Exp-6/Run-7)
Damping Ratio
0.00
0.02
0.04
0.06
0.08
0.10
0.12
0.14
0.16
0.18
40
60
80
100
120
140
Temperature (Deg.C)
40
60
80
100
120
140
Temperature (Deg.C)

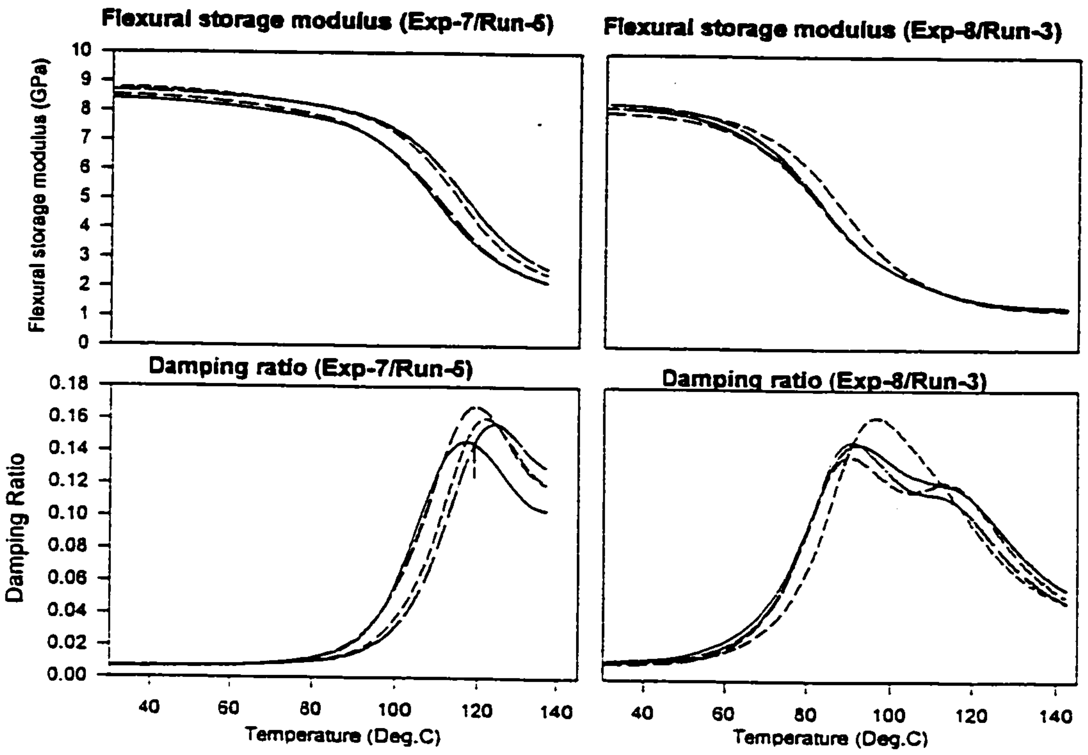
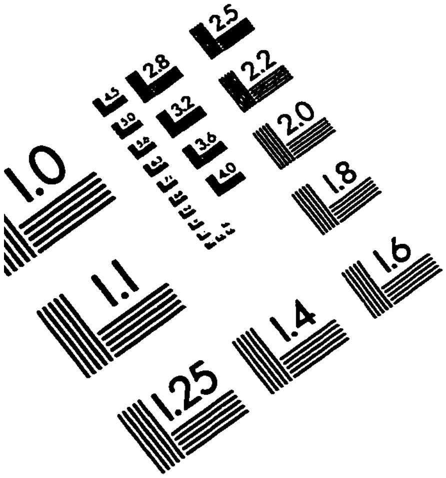
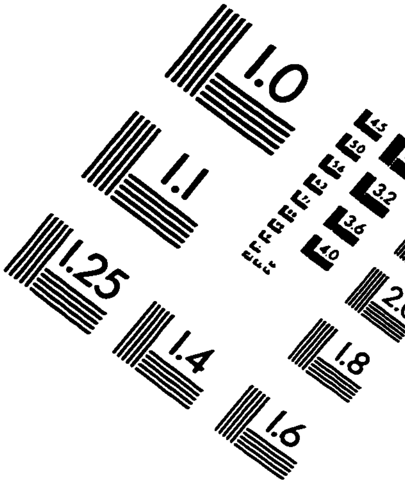
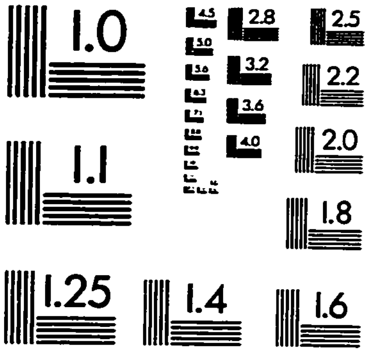
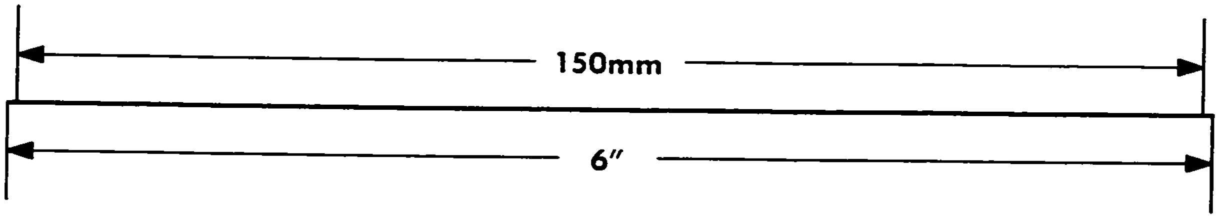

Flexural storage modulus (Exp-7/Run-5)
Flexural storage modulus (Exp-8/Run-3)
Flexural storage modulus (GPa)
0
1
2
3
4
5
6
7
8
9
10
Damping ratio (Exp-7/Run-5)
Damping ratio (Exp-8/Run-3)
Damping Ratio
0.00
0.02
0.04
0.06
0.08
0.10
0.12
0.14
0.16
0.18
40
60
80
100
120
140
Temperature (Deg.C)
40
60
80
100
120
140
Temperature (Deg.C)

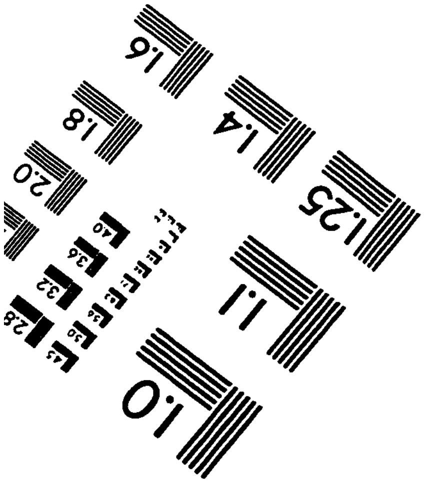
IMAGE EVALUATION
TEST TARGET (QA-3)











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