MECHANICAL AND MICROSCOPY ANALYSIS OF CARBON FIBER REINFORCED POLYMERIC MATRIX COMPOSITE MATERIALS

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The report is prepared in cooperation with the State of Florida Department of Transportation and the Federal Highway Administration.

ACKNOWLEDGMENTS

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1. ABSTRACT

This is the final report for a proposal funded by the Department, of Transportation to investigate the' mechanical properties of the bonding material. (resin/epoxy) in relation to the durability of Carbon fiber reinforced polymeric matrix composites (FRP) for various temperature conditions. In this research the intention was to measure the mechanical properties of the resin and its bonding characteristics with Carbon fiber in order to investigate its temperature dependence. Investigation of the interface properties will require both mechanical testing (shear' testing) and microscopy (optical; and electron microscopy) techniques to examine the redistribution of stresses in the epoxy resin material and its effect on the interfacial strength. The effect of temperature in the range of -20°C to 600C were also carried in this investigation by performing the appropriate tests. The microscopy analysis were achieved by using an ESEM Scanning Electron Microscope. A micro-tensile stage was constructed as result of this project. An important component of the:

1.1 Background

Structural composites possessing superior strength/weight and modulus/weight characteristics including high performance fiber (glass, carbon, aramid and hybrid) reinforced composites with polymer matrix are being considered as alternative materials by the Department of Transportation to reinforce concrete and also to repair prestressed concrete damaged by overweight vehicle impact. Composite materials are being used and manufactured as reinforcing materials in the design of structures in a variety of other applications. Conventional steel reinforced structures (e.g., bridges) are exposed to marine environments that causes serious deterioration due to corrosion. This problem may be eliminated by using advanced composite FRP materials as reinforcing elements due to their excellent corrosion resistance under- humid, salt water and ultraviolet conditions. These results, however, lack some important information (e.g., changes in the elastic and plastic properties at some operating temperatures) which are used in the design of structures [4],

The construction of these composites for specific tasks requires the use and selection of the two, fiber and epoxy resin, constituents. A large amount of research has been invested to manufacture the best fiber material possible. With the emergence of super quality carbon fibers the problem has been reduced to find and select the best epoxy resin possible. The problem becomes even more complicated once a third component or fiber/epoxy resin interface is introduced. The durability and usefulness of the composite depends highly on the strength of these interfaces. Also their mechanical properties under different environmental conditions are not well understood and/or documented [4].

The construction of these composites for specific tasks requires the use and selection of the two, fiber and epoxy resin, constituents. A large amount of research has been invested- to manufacture best fiber material possible. With the emergence of super quality carbon fibers the problem has been reduced to find and select the best epoxy/resin possible. Two types of resins were examined in this research (provided by the DOT research). Investigation of the interface properties require both mechanical testing (shear testing) and microscopy (optical, and electron microscopy) techniques to examine the redistribution of stresses in the epoxy resin material and its effect on the interfacial strength. The effect of temperature in the range of -20°C to 60°C were also considered in this investigation. The problem becomes even more complicated once a third component or "fiber/epoxy resin interface" is introduced. The durability and usefulness of these composites depends highly on the strength of these interfaces. Also their mechanical properties under different environmental conditions are not well understood and/or documented.

1.1 Objective / Scope of the Work

The goal of this proposal is to investigate the mechanical properties of the bonding material (resin/epoxy) in relation to the durability of the fiber reinforced polymeric matrix composites (FRP) for various temperature conditions. Specifically we will concentrate on carbon fiber-FRP's. In this proposal, mechanical properties of the resin and its bonding characteristics with Carbon fiber and Concrete will be investigated to arrive at a unified durability model. This model will include geometry, temperature and water moisture as its parameters, as well as those already mentioned.

In the first phase of the project:

l- The design of a micro-tensile stage was completed. The final design and schematics of the construction is included in the Appendix.

2- A complete study has been performed on composite models including the effects of hold time, nonlinearity, and rate dependency. It is intended to use these models to develop a durability model for the second year of the project.

3- A survey has been performed on fire protecting barrier coating materials.

In the second phase of the project:

1- The measurements of the tensile strength of the composite and the- bonding strength of the composite/composite specimens have completed at room temperature and presented in the following chapters.

2- The construction of the micro-tensile stage has been completed. At this point the microtensile stage can operate at very low load ranges (less than 100 lb.) and requires a total modification in design to be useful for the type of materials dealt in this proposal.

3- A table top testing machine has been reconfigured to test fibers for their strength. Initial results showed that an intensive investigation is required to find better gripping configurations to determine the bonding strength of individual fibers.

4- A complete study has been performed on composite models including the effects of hold time, nonlinearity, and rate dependency. It is intended to use this model` to develop a durability model for the second stage of the project.

In the final phase of the proposal:

1- A high temperature facility was used and configured to test the mechanical properties of these composites (Elastic Modulus, strength, and ductility).
2- A microscopic examination of the composite materials was performed and the volume fraction of the fiber/matrix was measured and correlated to the mechanical strength.

A major part of this project was consumed in the preparation of specimens for the proper experiments. According to the recommendations given by the department of transportation, it was important to prepare specimens to simulate the field application of these materials.   In the ideal situation the composite materials are produced using an autoclave' in order to avoid any defects and also the least amount of air trapped inside the material. As a result the results for the mechanical characterization reflect the range of errors involved in the use of these materials. Specimens have been prepared for the fiber; epoxy resin and the carbon fiber reinforced composite materials. All the tests at room temperature up to 60°C have been completed and will be presented in the following chapters. The creep properties of the resin materials have been measured for this temperature range in this phase of the research. It is intended to prepare tensile and microscopic and expose them to different temperature conditions inside the chamber of an Environmental Scanning Electron Microscope (ESEM). The microtensile stage developed is incapable of introducing the required amount of load on the composite specimen for a representative segment of the material. We are in the process of developing a high capacity (1000 lb.) microtensile stage to produce the required loads on the composite specimens. The mechanical testing experiments were performed on the Materials Testing Machine (MTS) and the microscopy analysis were performed using the Environmental Scanning Electron Microscope and optical microscope. All tests were carried out under the supervision of Dr. Shahawy from the Structural lab of the Department of Transportation.

CHAPTER 2

LITERATURE REVIEW

2.1. Composite Materials

A composite material is a manufactured material consisting of two or more materials combined on a macroscopic scale to form a useful material. The key is the macroscopic examination of the material. Different materials can be combined on a microscopic scale but, the resulting material is macroscopically homogeneous. Composites are more advantageous because they usually exhibit the best qualities of their constituents and often some that neither possesses. The properties that can be improved by forming a composite material include: strength, stiffness, corrosion resistance, water resistance,: weight, fatigue life, temperature-dependent behavior, thermal insulation, and thermal conductivity [1].

There are three common types of composite materials:

       1. fibrous composites - consist of fibers in a matrix

       2. laminated composites - consist of layers of various materials

       3. particulate composites - composed of particles in a matrix [1].

2.1.1 Fibers

Reinforcements come in a variety of forms. They can be in the form of long fibers, particles, flakes, whiskers, discontinuous fibers, continuous fibers, and sheets. It turns out that a great majority of materials are much stronger and stiffer in the fibrous form than in any other. These so-called advanced fibers posses very high strength and very high stiffness coupled with a very low density [2].

Fibers tend to have a more perfect structure in that the crystals are aligned in the fiber direction along the fiber axis. This geometry is crucial to the evaluation of their strength and must be considered in structural applications. An example being carbon with a density of only 2.268 g/cm^3. Arranged in

the graphitic crystalline structure, the carbon atoms are arranged in the form of hexagonal layers anc are anisotropic with a Young's modulus in the layer plane being equal to about 1000 GPa. Along the axis perpendicular to the layer plane Young's modulus is only about 35 GPa. The graphitic structure has a very dense packing in the layer plane. Thus, the high-strength bond between carbon atoms it the layer plane results in an extremely high modulus while the weak van der Waals type bond between the neighboring layers results in a lower modulus in that direction: Consequently, this orientation of hexagonal planes along the fiber axis allows these fibers to possess very high strength and stiffness with very low density [1].

The use of fibers as high-performance engineering materials is based on three important characteristics:

1. A small diameter with respect to its grain size or other microstructural unit. This allows a higher fraction of the theoretical strength to be attained than that possible in a bulk form. This is a direct result of the so-called size effect; that is, the smaller the size the lower the probability of having imperfections in the material.

2. A high aspect ratio (length/diameter, 1/d) that allows a very large fraction of the applied load to be transferred via the matrix to the stiff and strong fiber:

3. A very high degree of flexibility that is really a characteristic of a material having a high modulus and a small diameter. This flexibility permits a variety of techniques to be employed for making composites with these fibers [2].

2.1.2 Matrix

Naturally, fibers are of little use unless they are bound together to take the form of a structural element which can take loads. This binder material-is called a matrix. The purpose of the matrix is manifold: support, protection, stress transfer, etc. Typically, the matrix is of considerably lower density, stiffness, and strength than the fibers. However, the combination of fibers and a matrix has proved to have a very high strength and stiffness, yet still have low density [1].

Epoxy resin, a common polymer matrix, has many advantageous properties. It has adequate resistance to water and a variety of chemicals, weathering, aging, and can withstand temperatures up to about 80-C. Its very low cost is an added bonus [2].

2.1.3 Interfaces

To obtain desirable characteristics in a composite material, the applied load should be effectively transferred from the matrix to the fibers via the interface. The interface consists of near surface layers of fiber and matrix and any layer(s) of material existing between these surfaces. The interface is of great importance because the internal surface area occupied by the interface is quite extensive. It can easily go as high as 3000 em^2/cm^3 in a composite containing a reasonable fiber volume fraction [2].

The important types of interfacial bonding are as follows:

Mechanical bonding

Simple mechanical keying effects between two surfaces can lead to a considerable degree of bonding. Any contraction of the matrix onto a central -fiber would result in a gripping of the latter by the former. Pure mechanical bonding alone is of enough. However, mechanical bonding could add, in the presence of reaction bonding, to; the overall bonding. Also, it is efficient in load transfer when the applied force is parallel to the interface.

Chemical bonding

There are two types of chemical bonding:

1. Dissolution and Wettability Bonding.   Interaction between components occurs at an electronic scale. Since these interactions are of rather short range, it is important that the components come into an intimate contact on an atomic scale. This implies that surfaces should be appropriately treated to remove any impurities. Any contamination of fiber surfaces or entrapped air or gas bubbles at the interface, will hinder the required intimate contact between components.

2. Reaction Bonding. A transport of atoms occurs from one or both of the components to the reaction site, or the interface. This atomic transport is controlled by diffusion processes. Two polymer surfaces may form a bond owing to the diffusion of matrix molecules to the molecular network of the fiber, thus forming tangled molecular bonds at the interface [2].

2.2   Micromechanical Behavior of Composites

In most structural design, most laminated composite structures are designed for fiber-dominated load paths. Despite this criterion, a need exists for a fundamental understanding of the stress/strain relationships of laminates that are loaded along matrix-dominated directions. This need arises from three possible sources. The first is the desire for analytical methods to perform local stress analysis of structural components (e.g. discontinuities, stress concentrations, mechanically, fastened joint, failure zones) which may lack fibers along the primary load path. In this research we have concentrated on matrix dominated models since in the long term life it is the matrix (the resin) which plastically deforms or creeps and finally fails. Cracks also usually propagate through the matrix.

For a complete durability model, long-term exposure to load and temperature may need to be accounted for to accurately predict the effects' on the time-independent and time-dependent mechanical behavior of the matrix-dominated polymer matrix composites. we will include a complete review and our final form of the model in the second quarterly report. At this point it is sufficient to mention that the correct modeling effort is totally dependent on the initial result of our creep tests.

The mechanics of materials approach uses simplified assumptions regarding the mechanical behavior of -a composite material. The most prominent assumption is that the strains in the fibers' axial direction of a unidirectional fibrous composite are the same in the fibers as in the matrix as shown in Figure 1.
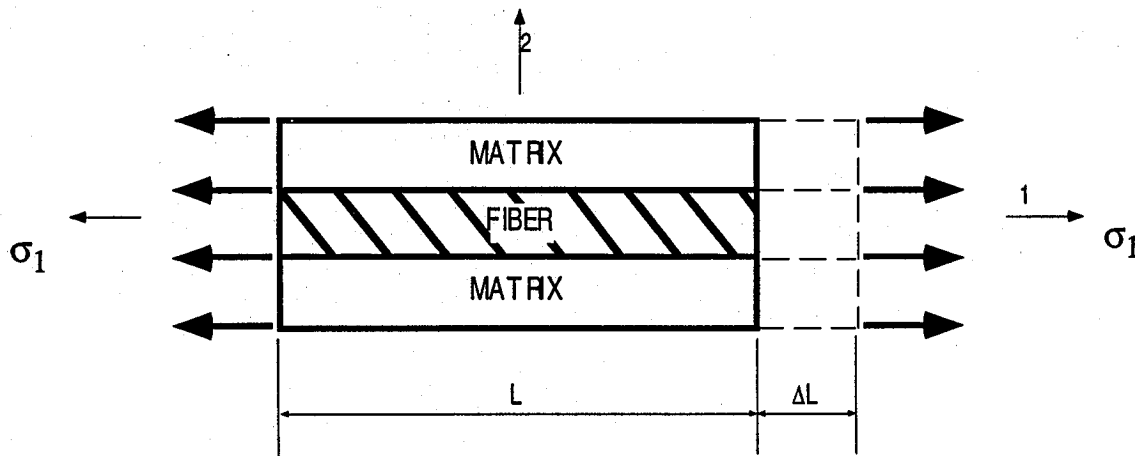
Figure 1. The fiber matrix arrangement for an element of laminated composite

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The four properties of such a composite are:

1. Young's modulus (modulus of elasticity) of the composite in the direction of the fibers, defined as E_1

$$E_t = E_f V_f + E_m V_m$$

(1)

This is known as the rule of mixtures, an expression for the apparent Young's modulus in the direction of the fibers.

2. Young's modulus of the composite in the transverse direction, E_2

$$E_2 = \frac{E_f E_m}{V_m E_f + V_f E_m}$$

(2)

3. Major Poisson's ratio, defined as
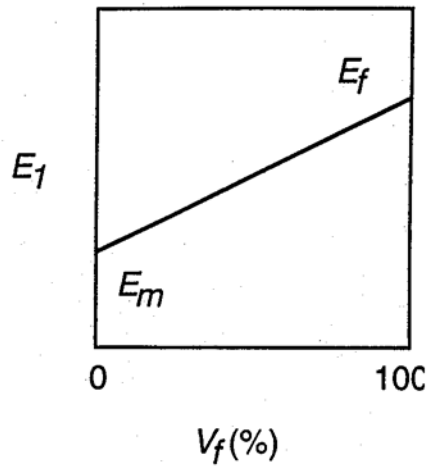
$$v_{12} = \frac{\varepsilon_2}{\varepsilon_1}$$

(3)

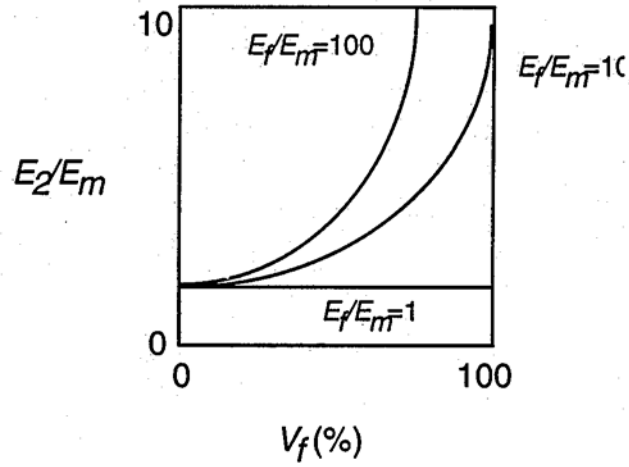$$V12 = V_m V_m + VFV f$$

(4)

4. In-plane shear modulus, G12

$$G_{12} = \frac{G_m G_F}{V_m G_f + V_f G_m}$$

(5)

The variations of E_1, E_2, V_{12}, and G_{12} with fiber volume fraction are illustrated in Figures $2 - 5$.

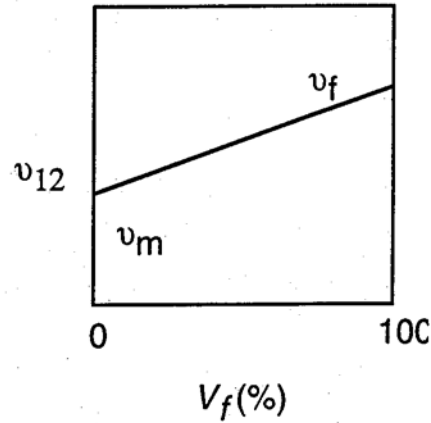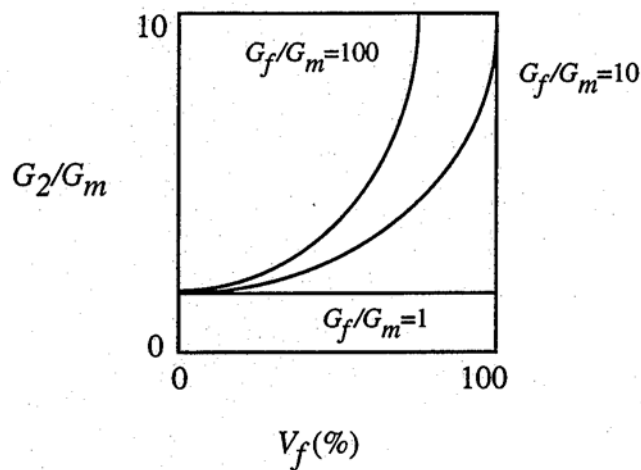**Figure 2.** Variation of E_1 with fiber volume fraction

Figure 3. Variation of E_2 with fiber volume fraction

Figure 4. Variation of v_{12} with fiber volume fraction

Figure 5. Variation of G_{12} with fiber volume fraction

Following the mechanics of materials approach, it is clear that a greater volume fraction of fibers will yield a stronger and stiffer composite. Therefore, a logical conclusion would be to use 100% fibers and no matrix. However, this is impossible -because the matrix is what provides support and protection to the fibers, as well as a medium through which the stress is transferred. Thus, an ideal fiber to matrix volume ratio must be determined [3].

CHAPTER 3

MATERIALS TESTED

3.1 Carbon Fiber

Thornel Carbon Fiber T-300 12K is a continuous length, high strength, high modulus fiber consisting of 12,000 filaments in a one-ply construction. The fiber surface has been treated to increase the interlaminar shear strength in a resin matrix composite. The typical properties of this composite are listed in Table 1.

Table 1. Typical Properties of Thornel Carbon Fiber T-300 12 K.

Property	Value
Tensile Strength	3.65 GPa
Tensile Modulus	231 GPa
Filament Diameter	7 μm
Density	1.76 Mg/m^3
Elongation at Failure	1.4%
Surface Area	0.45 m^2/g

3.2 Composite Matrix

Two types of epoxies are used as the matrix of these composites which are introduced in the following.

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3.2.1 Modified Amine Epoxy

This epoxy is manufactured by the Daubert Chemical Company, Inc. under the brand name of Daubond DC 6323. It is a general purpose, two-part epoxy adhesive. It is color-coded for identification and mixing and cures at room temperature. Also, it possesses good adhesion to various substrates: wood, glass, concrete, metal, and some plastics.

Table 2. Typical properties of the modified amine epoxy.

	DC 6323 "A"	DC 6323 "B"
Component	epoxy resin base	curing agent
Color	white	black
Viscosity	15,000-25,000	25,000-35,000
Mix ratio by volume	1	1
by weight	110	110
Density	9.6 ± 0.2 lbs/gal	10.6 ± 0.2 lbs/gal
Pot life (4 oz.)	1-2 hours	1-2 hours

3.2.2 Thixotropic Epoxy

This epoxy is manufactured by PTM&W industries, Inc. under the brand name PH2032. It is a medium viscosity, unfilled, light amber laminating resin that is designed for structural production applications. This resin laminates very easily, and wets out fiberglass, carbon, and aramid fibers readily. Used with PH3660 hardener, this system cures at a minimum temperature of 72-F and should be cured at least 24 hours before moving the, structure. Typical properties are listed in Table-3.

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Table 3. Typical properties of thixotropic epoxy.

	PH2032	**PH3660**
Component	epoxy resin base	curing agent
Color	light amber	amber
Viscosity	3700 cps	250 cps
Mix ratio by volume	3.2	1
by weight	100	27
Density	9.51 lbs/gal	8.34 lbs/gal
Pot life (4 oz.)	---	50-60 min.

3.3 Fire Retarding Materials (FRM):

Every combustion .and burning process requires a certain activation energy. In other words, for each flammable material a certain amount of heat is required until the material burns spontaneously. In principle, we can add to the composite material , Chemicals (powders, ..) that require a high activation energy and will therefore not easily burn. Or, we can add a material that requires a certain activation energy to decompose into non-harmful volatile particles. An evaporation process requires an endothermic energy change and does not have enough activation energy that would be needed to initiate the burning process. An initial study showed that an addition of 20% vol. fraction of FRMs have improved not only the fire resistance but also a slight increase in strength also occurred. The resin material stopped burning as soon as the fire source was cut off. In the case of resins with no FRM, exposure to a fire source (torch) started a self-combustion flame which continued without the need for the source itself. Even though these results are encouraging but it shows that other materials are needed to be considered and probably invented for a better fire protection.

Ex4 is an intumescent flame retarding powder produced by FRC Technologies, Inc., is a proprietary blend of polymeric phosphate, polyhydric alcohol, and polyamine. Ex-4 is a flame retarding powder that imparts flame retardancy to water-based latex coatings, solvent based resin coatings, caulks, mastics, and sealant. The product can also be added to various plastics and polyurethane foams as a flame retarding system. EX-4 is listed as having an average particle size of 3-5% on a 325 mesh screen.

CHAPTER 4

EXPERIMENTAL PROCEDURE

4.1 Testing Machine

A Tenius Olsen, 60 kip, servo-hydraulic, tensile testing machine was used for both tension and shear testing. A high temperature screw driven ATS machine was used for all the high temperature tests.

4.2 Manufacture of Composite Samples

We began by covering a flat working area with two layers of wax paper to prevent sticking by the epoxy. Two rectangles of carbon fiber material, approximately 24" x 18" in size, were cut from the large roll and about 400 mL of epoxy was prepared. Two layers of carbon fiber were used because the Department of Transportation applies two layers when working with this material. One-third of the epoxy was spread out evenly onto the wax paper in an area large enough for the first layer of carbon fiber material. The carbon fiber was then laid out over the epoxy and pressed down so that the epoxy seeped through and the fibers were saturated. This process was :repeated once more with a second layer. The remaining epoxy was spread out and the composite was covered with wax paper. A heavy cylinder was then used to roll out any air bubbles present within the layers. The composite was allowed to cure for at least 24 hours. The procedure is briefly shown in figure 6.

After curing, the composite sheet was marked and cut into 6" x 1" strips and 2" x 1" tabs. These strips and tabs were lightly sanded. The sanding was necessary because the composite material had a very smooth texture and the epoxy used in constructing the samples would not adhere very well. The tension composite samples consisted of one 6" x 1" strip and four 2" x 1" tabs. A 2" x 1" tab was attached to each end of the strip with its respective epoxy as seen in Figure 7.
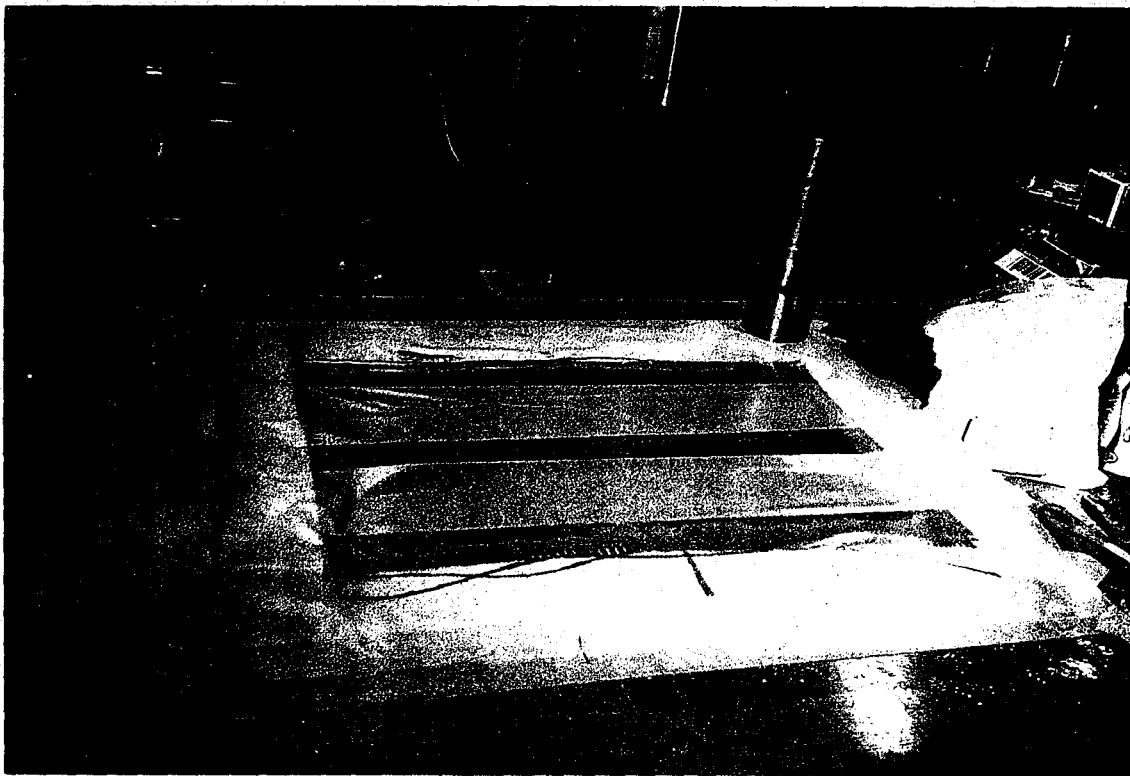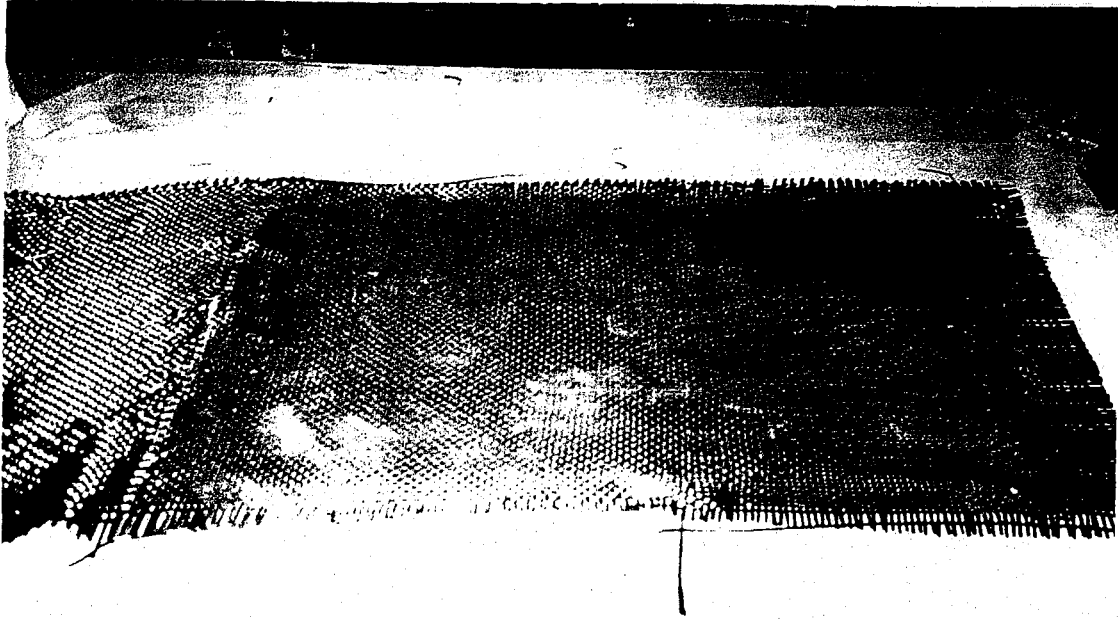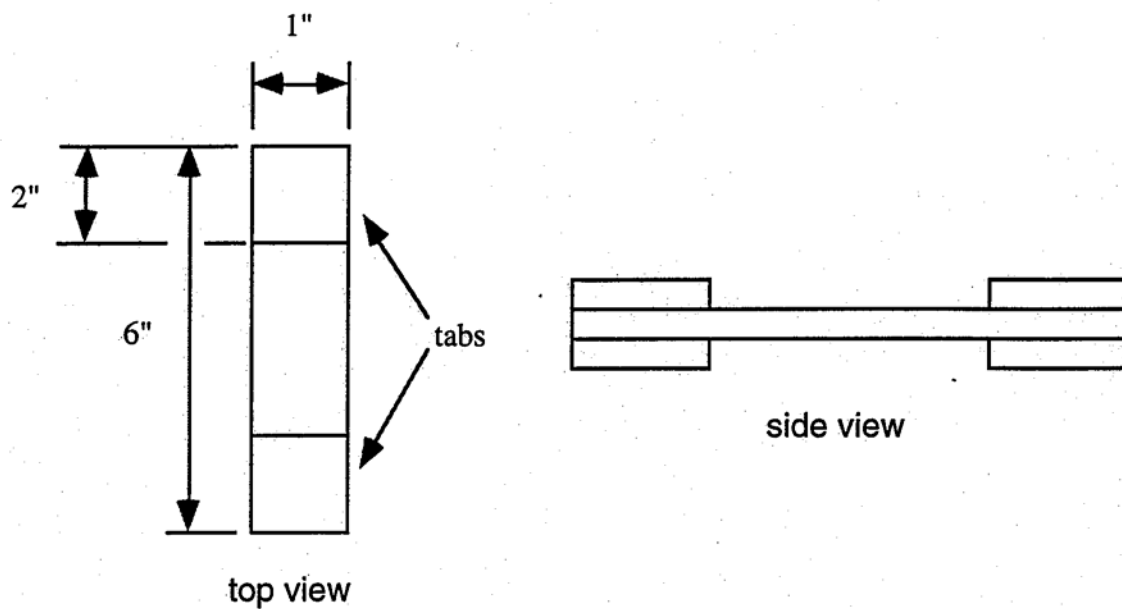
Figure 6.   The two stages of specimen

4.2.1 Tension Composite Samples

The tension composite samples consisted of one 6" x 1" strip and four 2" x 1" tabs. A 2" x 1" tab was attached to each end of the strip with its respective epoxy as seen in Figure 7.

Figure 7. Specimen-tab geometry for the composite sample preparation

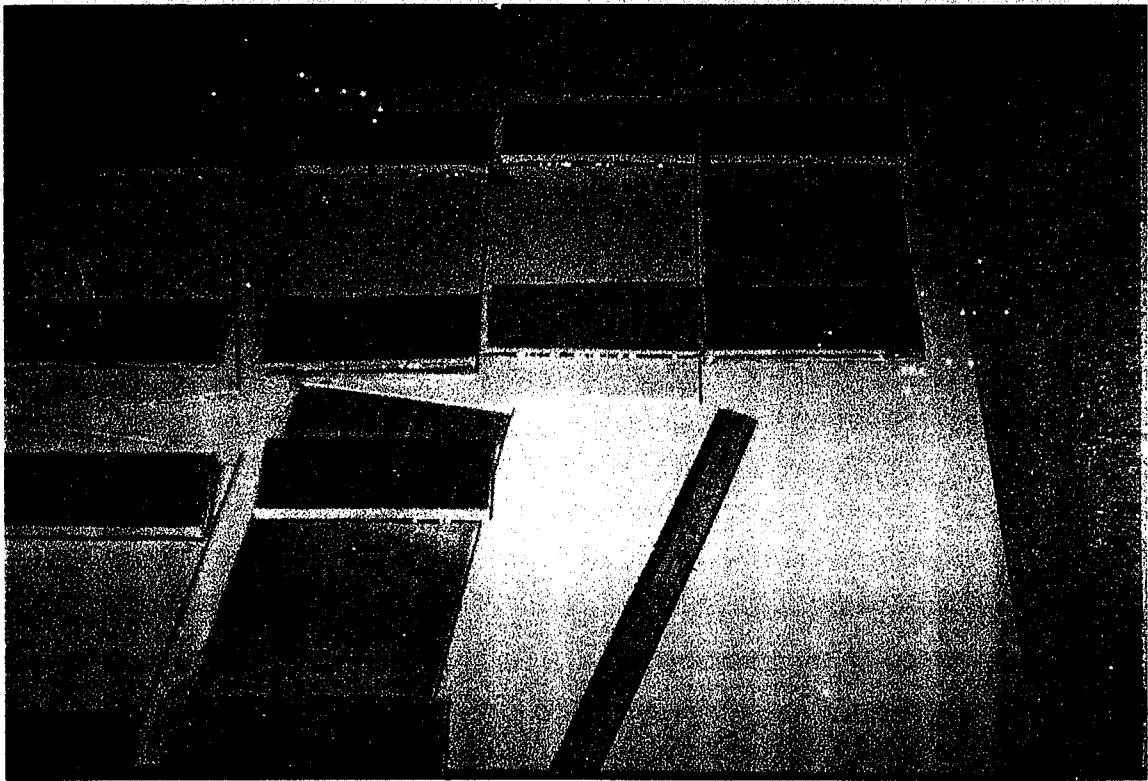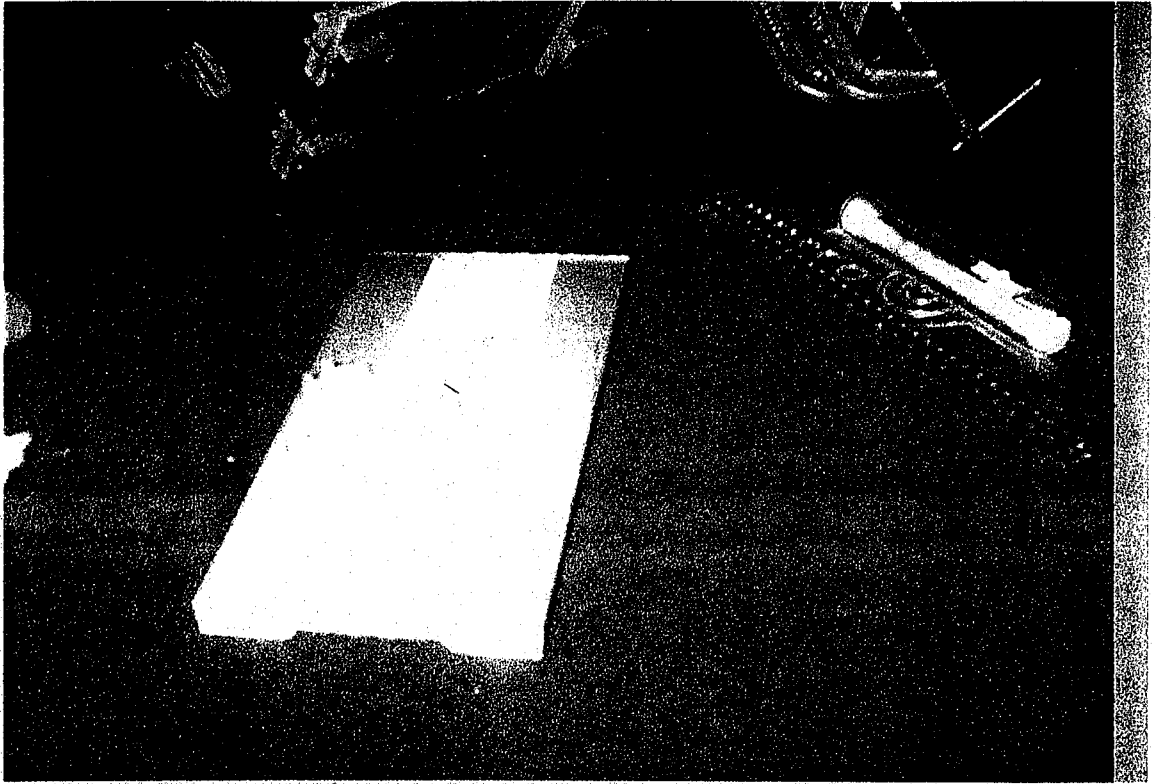The specimens are then cut using a special sawing machine as shown in Figure 8.

Figure 8. The location of the tab materials and the specimen preparation

4.2.2 Shear Composite Samples

The shear composite samples consisted of four 6" x 1" strips and four 2" x 1" tabs. They were attached together with epoxy as shown in Figure 7. The samples were designed to test the shear strength of the epoxy in between strips 1 & 2 and 3 & 4 (see figure) at mid length of the sample (overlap area) holding it together. Overlaps of 0.5" and 0.75" were used to determine an area that produced the best results. Also, the same epoxy used in making the composite was used in construction of the samples.
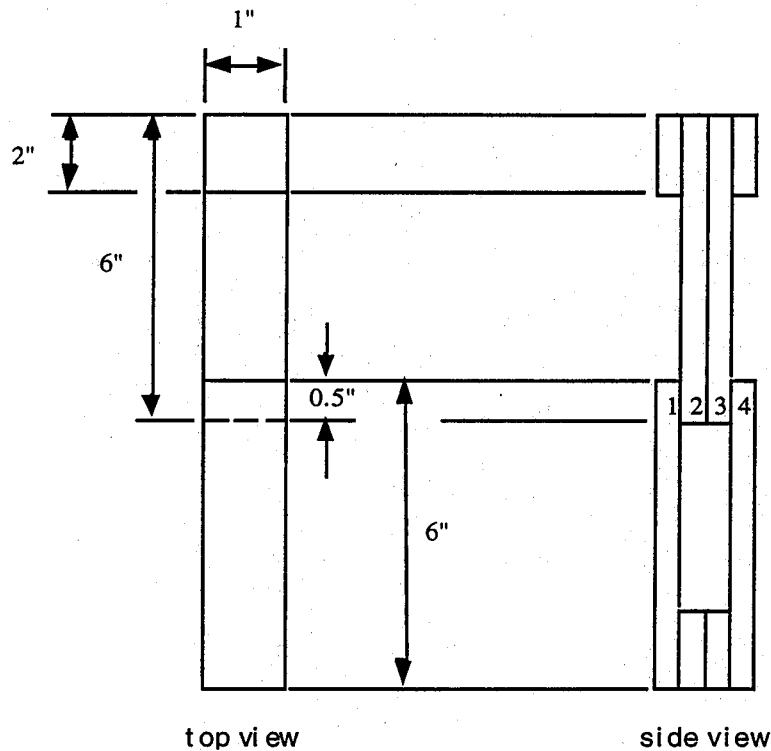
Figure 9. Schematic diagram for the shear lap specimens

4.3 Testing Procedure:

After the samples were allowed to cure for at least 24 hours at room temperature, they were tested for their shear and tensile strengths. Each sample was placed in the grips of the Tenius Olsen tensile testing machine. A constantly increasing force was applied until the sample failed.

CHAPTER 5

EXPERIMENTAL RESULTS

5.1 Room Temperature Experiments

5.1.1 Tensile tests

The tensile test was performed on five samples of each composite material made with modified amine epoxy and thixotropic epoxy. All samples were approximately the same width and length (= 1.16 x 6 inches) but because of the differing consistencies of the two epoxies the thixotropic samples were thinner(0.050 inches in thickness compared to 0.075 inches for the modified amine samples). The results of Tables 4 and 5 show that the Modified Amine samples were much stronger with an average tensile strength of 52.5 ksi as compared to the of 29.8 ksi for the Thixotropic samples.

Table 4. Tensile properties of composites (modified amine)

	Width (in)	Thickness (in)	Area (in²)	Load (lbs)	Strength (ksi)
1	1.150	0.05	0.058	3075	53.5
2	1.125	0.05	0.056	3180	56.5
3	1.150	0.05	0.058	3070	53.4
4	1.200	0.05	0.060	3150	52.5
5	1.150	0.05	0.058	2685	46.7
ave	1.155		0.058	3032	**52.5**

Table 5. Tensile properties of composites (Thixotropic)

Sam ple #	Width (in)	Thickness (in)	Area (in²)	Load (lbs)	Strength (ksi)
1	1.075	0.075	0.081	2355	29.2
2	1.225	0.075	0.092	2805	30.5
3	1.175	0.075	0.088	2600	29.5
4	1.150	0.075	0.086	2365	27.4
5	1.225	0.075	0.092	2955	32.2
ave	1.170		0.088	2616	**29.8**

5.1.2 Shear Test

The shear test was performed on two sets of samples. The first set was made up of five modified amine samples and five thixotropic samples with a shear area of approximately 1.16 inches by 0.50 inches (overlap). The second set was the same as the first except they had an overlap of 0.75 inches. The difference in overlap between the two sets was to determine the geometry of a sample that would produce optimum results. It turned out that the samples with an overlap of 0.75 inches produced inconclusive-data. The composite material failed intension before the epoxy failed in shear. This overlap proved to be too large. The samples with an overlap of 0.50 inches were successful. The thixotropic epoxy had a slightly larger shear strength than the modified amine. The samples with an overlap of 0.75 inches did not perform as expected but did demonstrate some interesting characteristics. The thixotropic samples could carry more load, but failed more catastrophically. Upon failure, the thixotropic samples -sent flying shrapnel through the air while the modified amine samples simply broke. The results of the shear tests are listed in Table 6, and 7.

**Table 6. Shear Properties of Composites (modified amine)**

	Width (in)	Overlap (in)	Area (in^2)	Force (lbs)	Shear Strength (ksi)
A	1.175	0.50	0.588	3280	2.8
B	1.150	0.50	0.575	3525	3.1
C	1.150	0.50	0.575	3600	3.1
D	1.200	0.50	0.600	1360	1.1
E	1.150	0.50	0.575	3485	3.0
Ave	1.165	0.50	0.583	3050	2.6

Table 7. Shear Properties of Composites (Thixotropic)

Sample	Width (in)	Overlap (in)	Area (in²)	Force (lbs)	Strength (ksi)
A	1.225	0.50	0.613	4465	3.6
B	1.125	0.50	0.563	4240	3.8
C	1.225	0.50	0.613	4160	3.4
D	1.150	0.50	0.575	3920	3.4
E	1.150	0.50	0.575	3800	3.3
Ave	1.175	0.50	0.588	4117	3.5

5.2 Varying Temperature Experiments:

These experiments were performed in a screw driven ATS machine. The set-up allowed a temperature variation from room temperature to 60 degrees. The results show a large variation in the data due to the inconsistencies in the specimen preparation. Figures 10-12 show the stress strain results for the three temperature ranges. The experiment was repeated for at least 10 different samples to provide a wide range of experimental data.
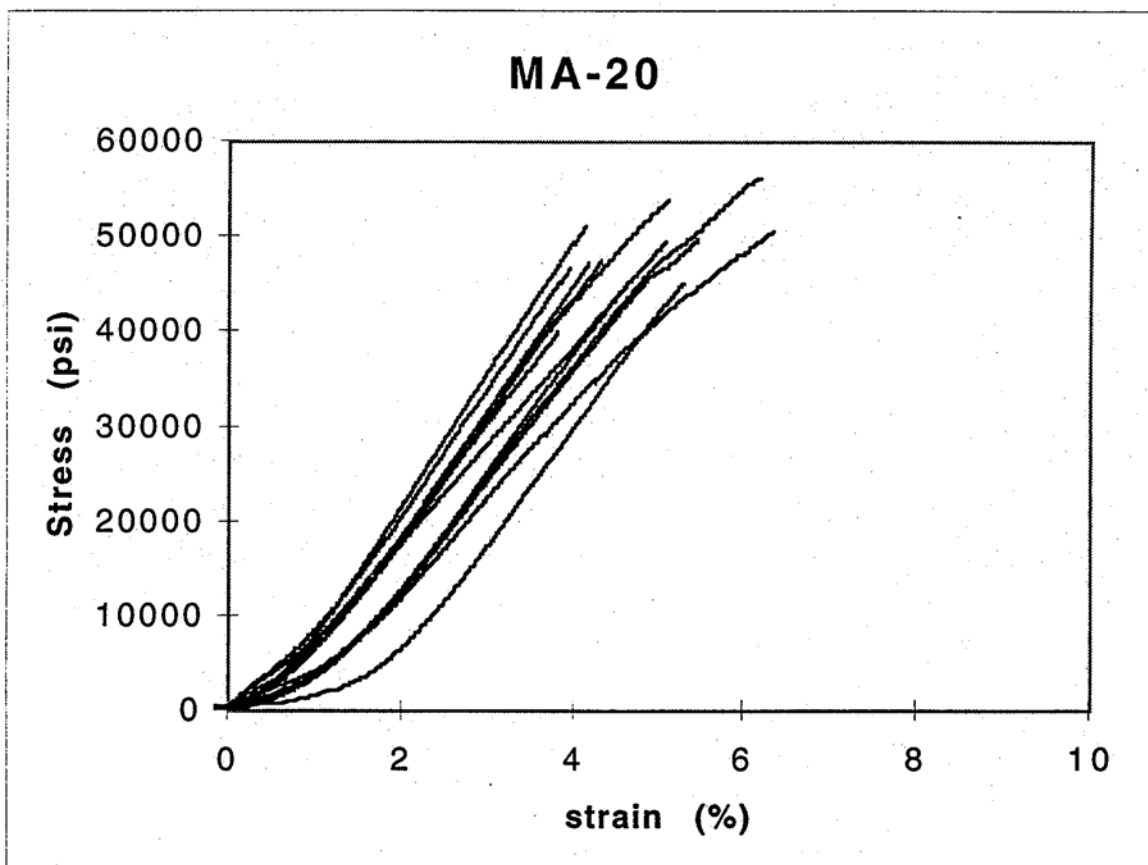
Figure 10. Stress-strain curve for mechanical testing of Modified Amine material at room temperature.
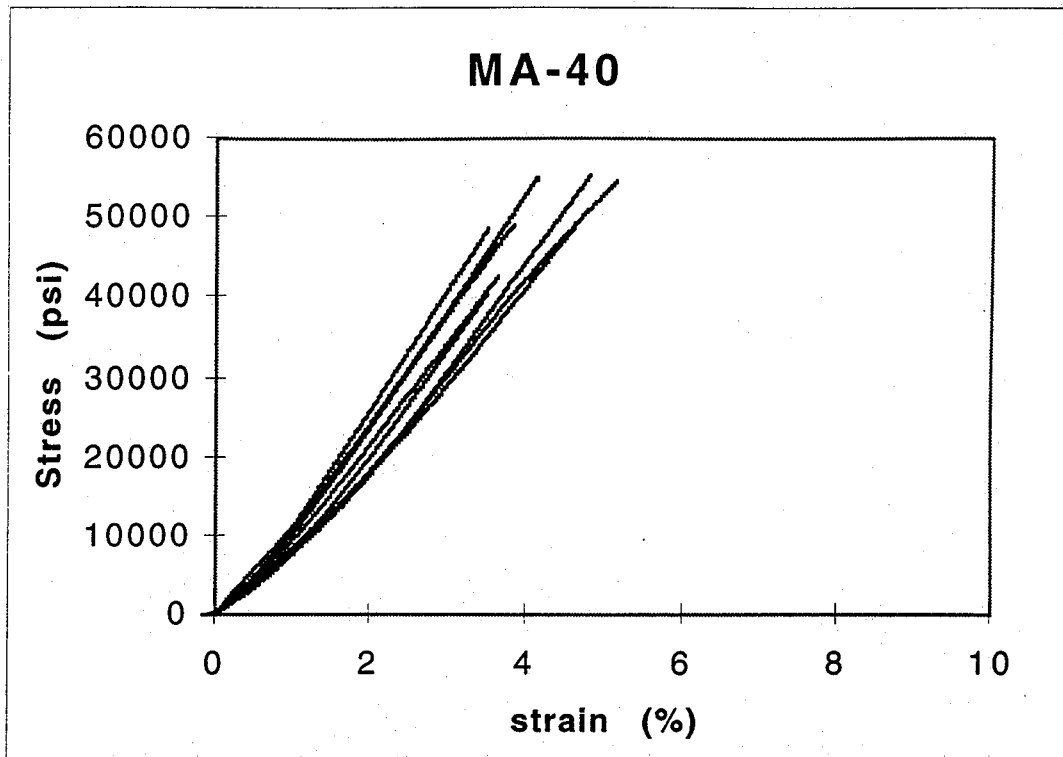
Figure 11. Stress-strain curve for Modified Amine material at 40 degrees C.
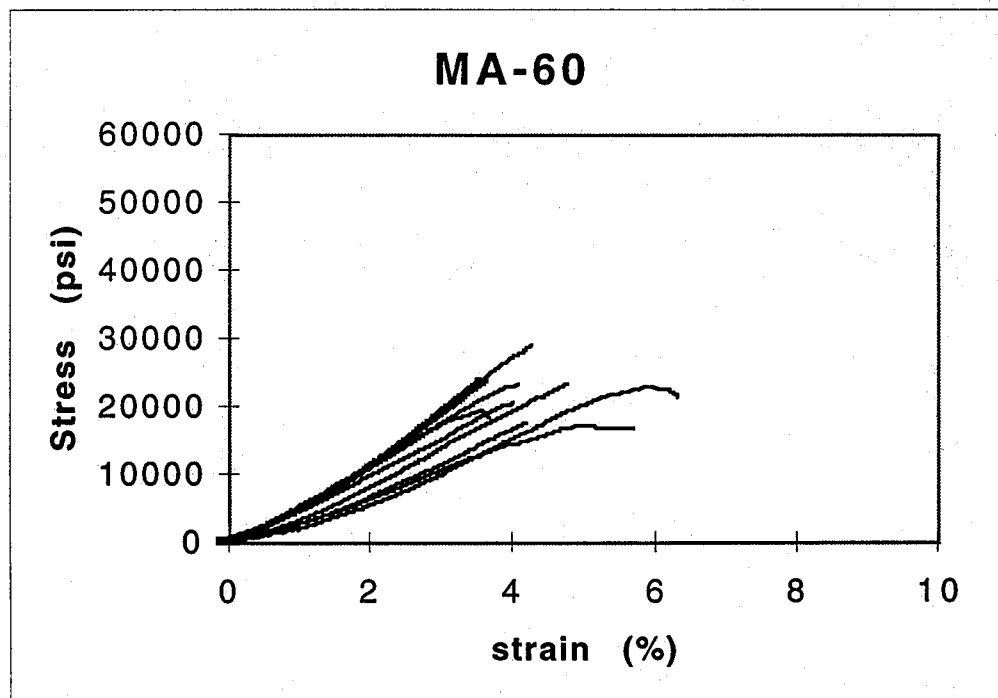
Figure 12. Stress-strain curve for Modified Amine material at 60 degrees C.
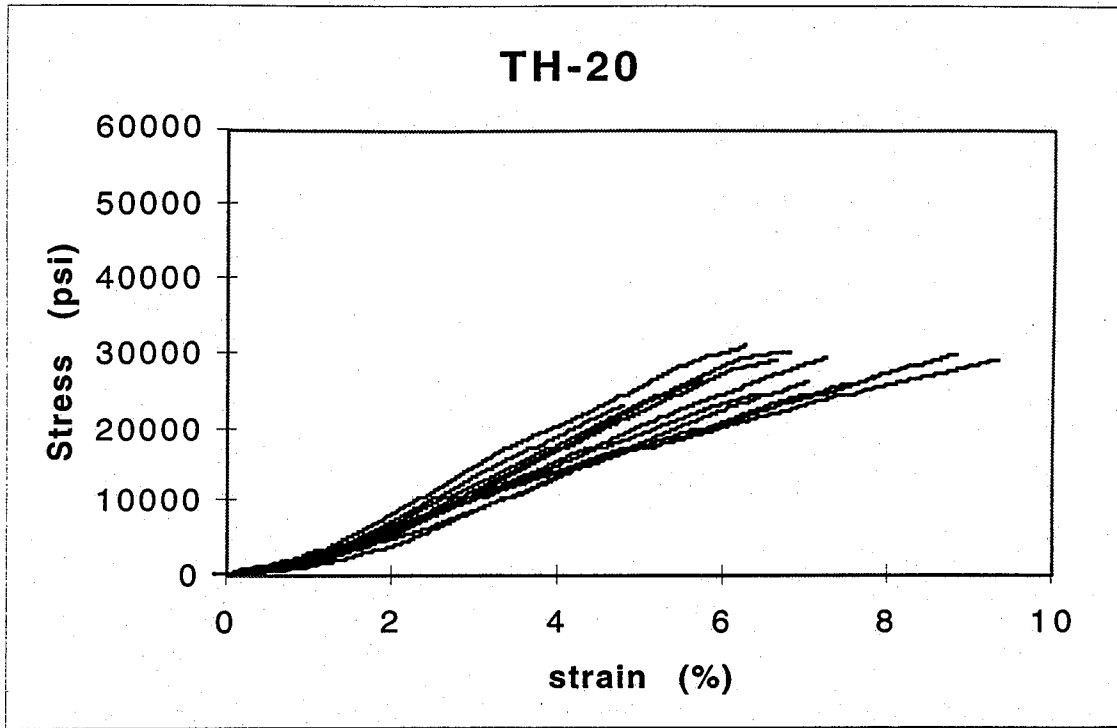
Figure 13. Stress-strain curve for Thixotropic material at 20 degrees C.
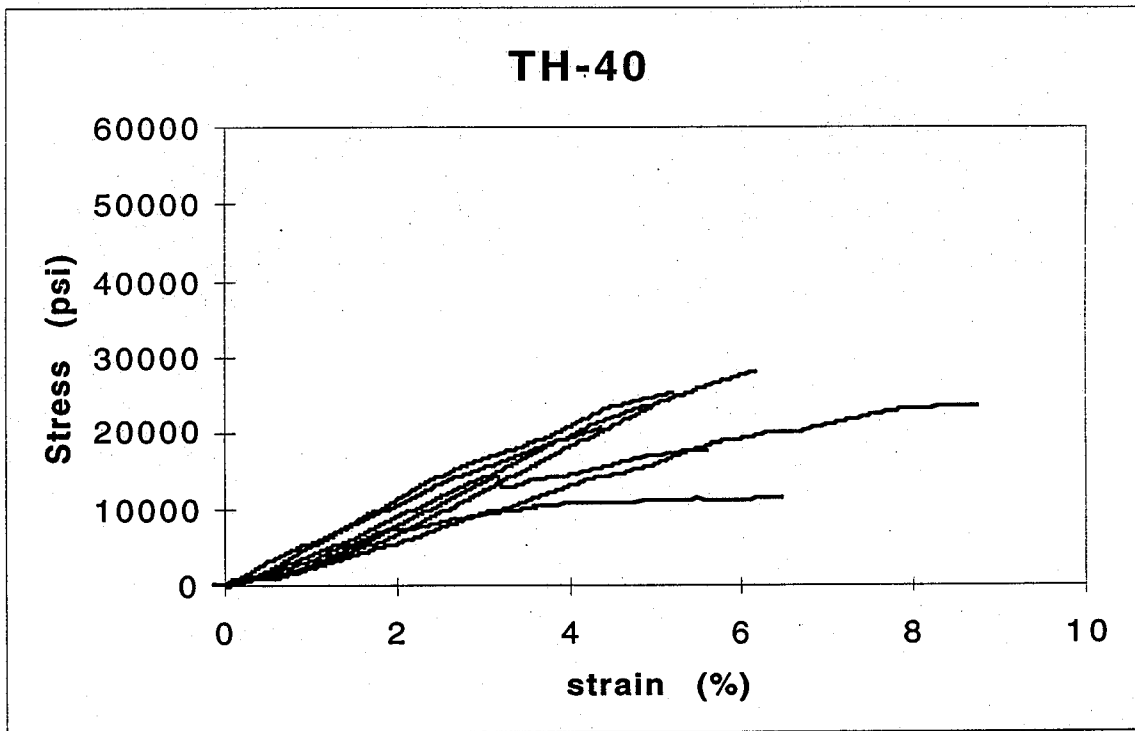
Figure 14. Stress-strain curve for Thixotropic material at 40 degrees C.
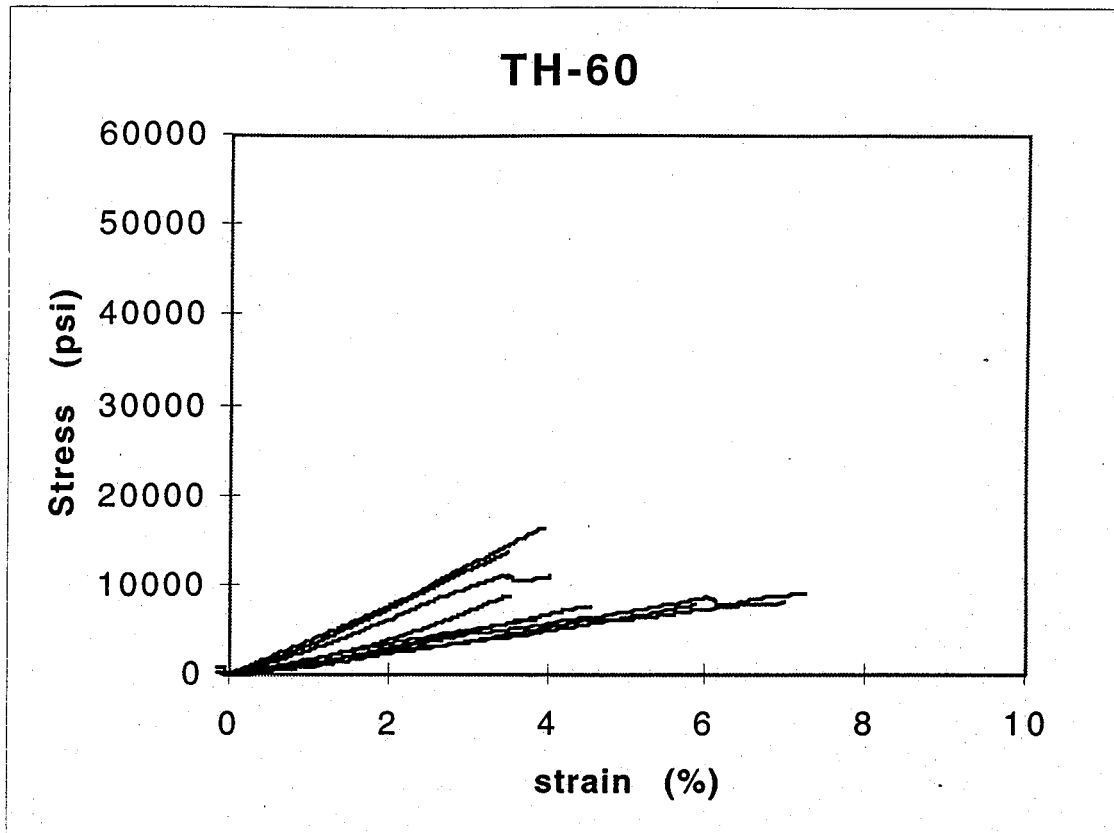
Figure 15. Stress-strain curve for mechanical testing of the Thixotropic material at 60 degrees C.

The result of the high temperature mechanical tests can be summarized in tables 8 and 9. The results show that both the strength and Elastic Modulus degraded with the increase in temperature. At 60 degrees the strength decreased by 50% in both cases. The elastic modulus decreased substantially for the case of Thixotropic material (from 0.5E6 psi to 0.1 E6). In the case of Modified Amine however a decrease of 40% was observed. The was no observable change in the ductility for the case of Modified Amine. The ductility however did reduce to approximately 50% in the -case of Thixotropic material.

28

Table 8. Mechanical Properties of Modified Amine material

Temperature (Degrees C)	Elastic Modulus (10^6 psi)	Strength (ksi)	Ductility (% strain)
Room	1.2-1.3	55	4-6
40	1.2-1.4	45-55	4-6
60	0.71	18-30	4-6

Table 9. Mechanical Properties of Thixotropic material

Temperature (Degrees C)	Elastic Modulus (10^6 psi)	Strength (ksi)	Ductility (% strain)
Room	0.42-0.6	25-30	6-9
40	0.37-0.55	18-28	5-8
60	0.1-0.4	8-16	4-7

5.3 Carbon Fiber Volume Fraction

To investigate the large variation in the experimental data Scanning Electron Micrographs of the Composite specimens were obtained using the ESEM environmental Microscope. Figures 16 and 17 are the result of this investigation for the two types of matrix materials. In the cross section of the carbon fiber composite the fibers that run both perpendicular and parallel to the cross section are visible as well as the epoxy material. The fibers appear as dark regions. Fibers running parallel to the cross section appear as light elongated strands that weave around the dark regions of perpendicular fibers. The epoxy makes up the remainder of the micrograph.

Figure 17 shows that there are clearly two separate regions in the matrix of the composite. . One region belongs to the fibers in the tensile direction of the material. Figure 18-a,b are the micrographs for this region for the two types of materials. Both figures 18a and b are obtained at a magnification of 10,100. Figure 18a is the micrograph belonging to the Modified Amine material and 18b is that of Thixotropic. It is obvious that there is a reaction zone between the fiber and the Thixotropic material. The fibers are not round and there exist a large amount of degradation at the fiber matrix interface. Two types of measurements were performed from these micrographs. From the large regions (Figure 17 at a magnification of 50X) the total volume fraction of the fibers in the loading (tensile direction was measured. From Figure 18 the localized volume fraction of the of these :fibers are found separately. An average volume fraction of 10% was measured as the total volume fraction of the fibers in the tensile direction. This means that the strength of the fibers have been reduced: (approximately) by 90%. This results in an average strength of 525 ksi for the Modified Amine and 300 ksi for the Thixotropic composite. The result, of this study is summarized in Table 10.

Table 10. Mechanical Properties of Thixotropic material

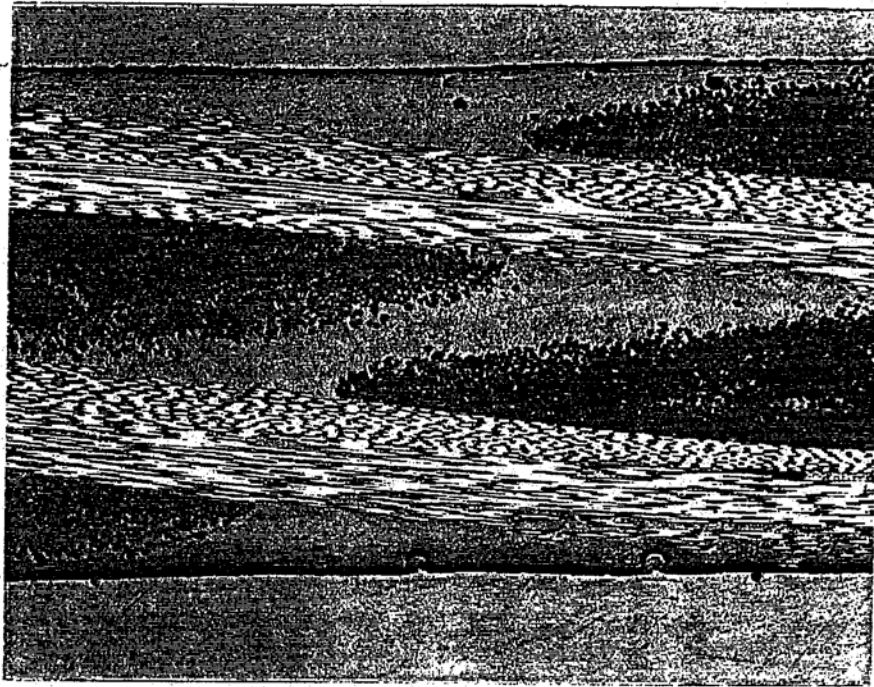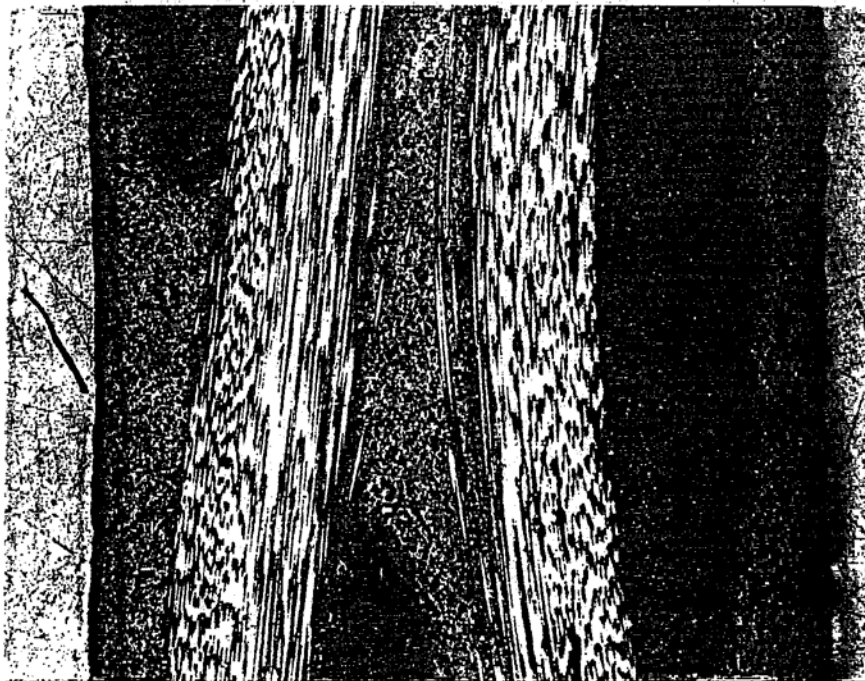| Epoxy Type	Global Area Fraction	Localized Area Fraction	Total Volume Fraction
Thixotropic	21.8%	44.1%	9.5%
Modified Amine	31.2%	34.0%	10.6%
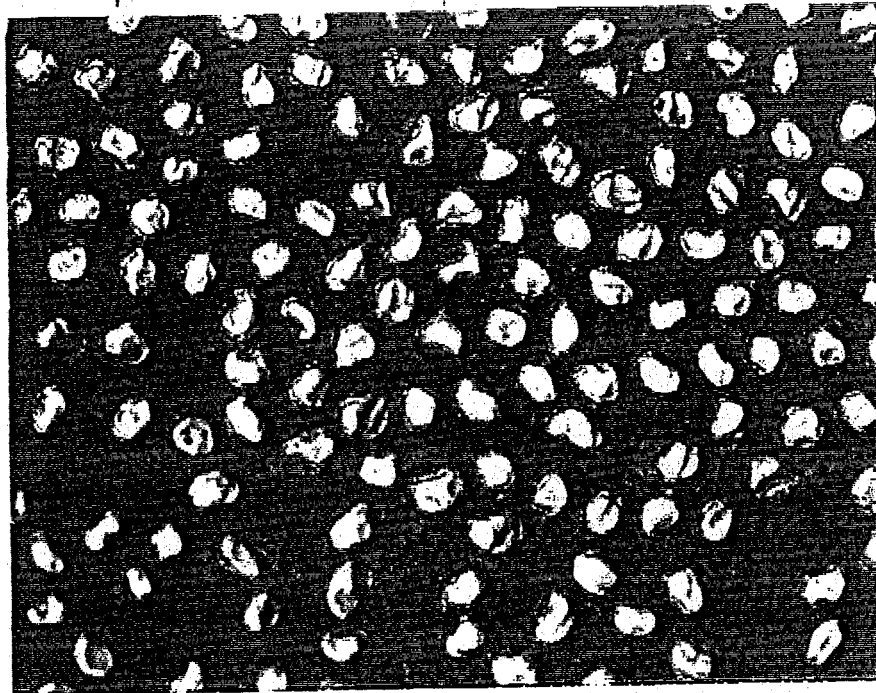
(a)   10,100X
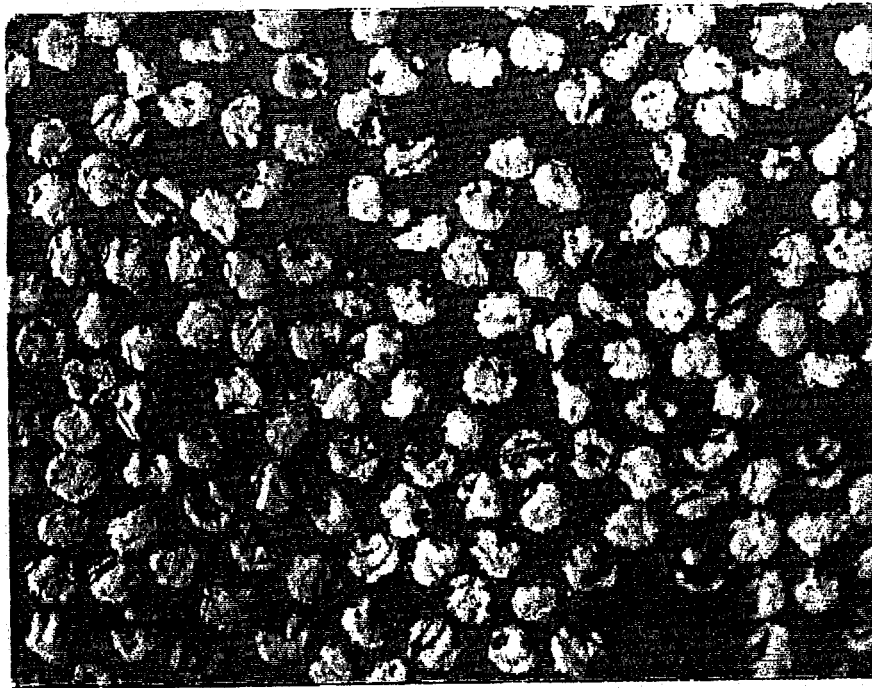
(b)   10, 100X

Figure 16- Scanning Electron Micrographs of a) Modified Amine and b) Thixotropic matrix composite material at a magnification of 10,100X.

Figure 17- Scanning Electron Micrographs of a) Modified Amine and b) Thixotropic matrix composite material at a magnification of 50x.

CHAPTER 6

CONCLUSION

The results showed that the Modified amine samples were stronger than the Thixotropic samples in both the shear and tension tests. The Modified Amine samples had an average tensile strength of 52 ksi and an average shear strength of 3.5 ksi, while the Thixotropic samples had an average tensile strength of 30 ksi and an average shear strength of 2.7 ksi. Although the Modified Amine samples proved to be stronger, extra care should be taken when using them in following experiments because of their tendency towards catastrophic failure. The tension sample and shear sample with an overlap of 0.5 inches proved to provide optimum results. The high temperature test results show that for both materials both the strength and Elastic Modulus degraded with an increase in temperature. At 60 degrees C the strength decreased by 50% in both cases. The elastic modulus decreased substantially for the case of Thixotropic material (from 0.5E6 psi to 0.1 E6). In the case of Modified Amine however a decrease of 40% was observed. There was no observable change in the ductility for the case of Modified Amine. The ductility however did reduce to approximately 50% in the case of Thixotropic material.
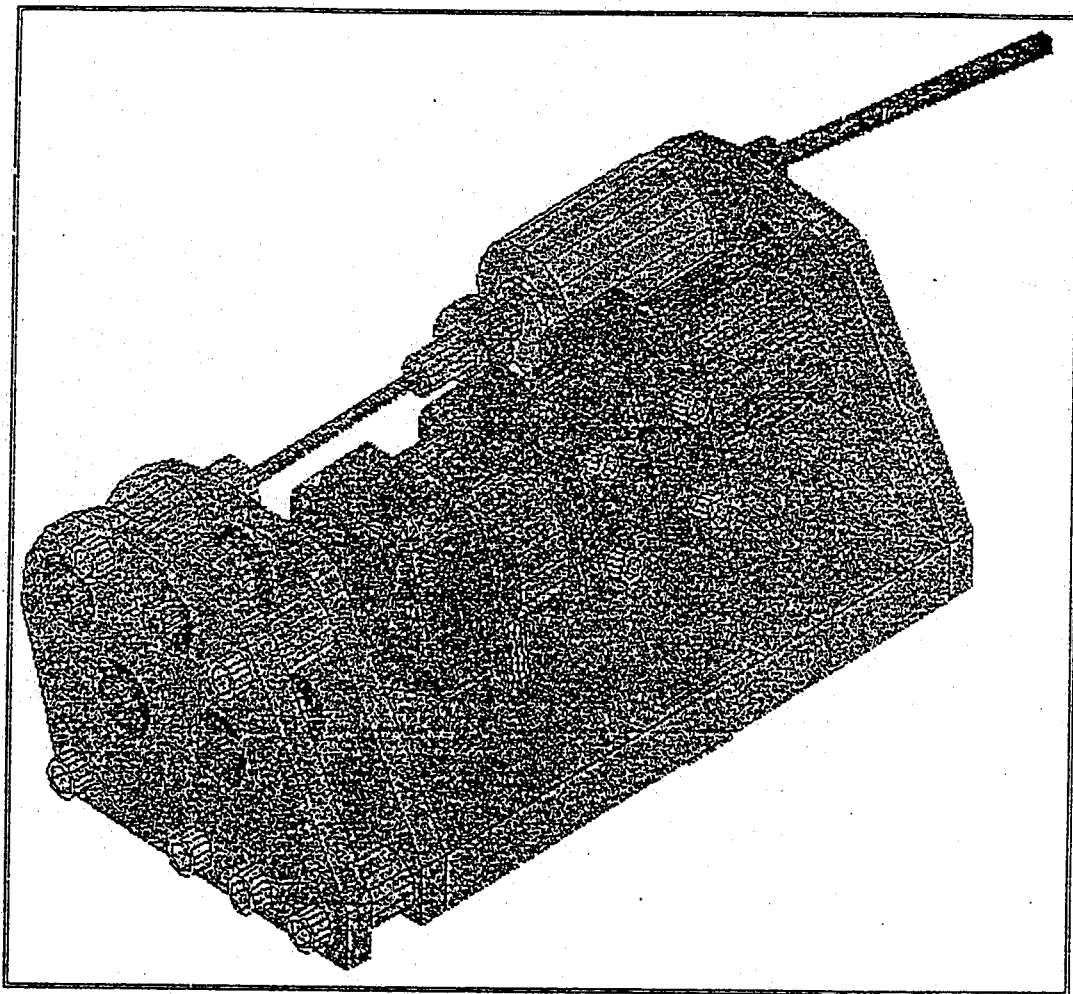
References

1. Robert M. Jones, *Mechanics of Composite Materials*, Hemisphere Publishing Corporation, New York (1975).

2. K. K. Chawla, *Composite Materials*, Springer-Verlag Inc., New York (1987).

3. A. Cox, J. Kuhlhoff and L. Lagorce, Manufacture and testing of glass composites at room and cryogenic temperatures., National High Magnetic Field Laboratory and FAMU/FSU College of Engineering (1993).

4. H. Garmestani, Mechanical and microscopy analysis of carbon reinforced polymeric matrix composite materials (proposal)., FAMU/FSU College of Engineering (1995).

Micro-Tensile Stage

For Electro Scan SEM

Contents:

Machining Inventory for the Micro-Tensile Stage:

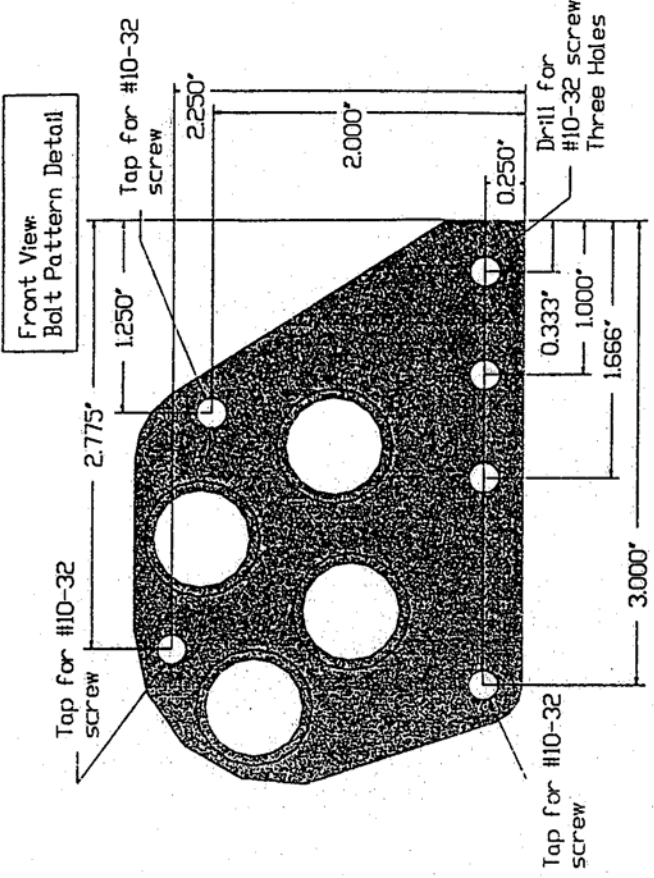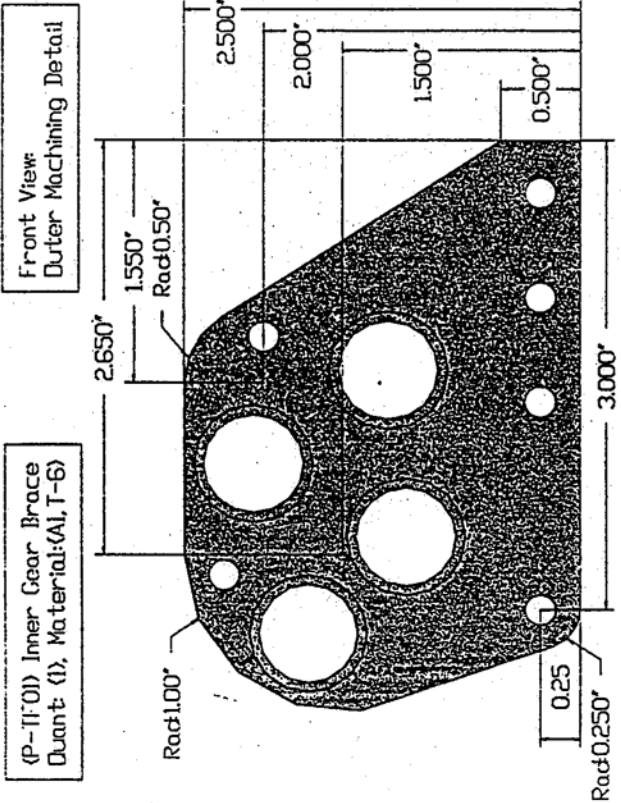| Part #	Part Name	Quantity	Description	Material
Tensile Stage Housing Parts:				
P-TF01	Inner Gear Brace	1	See Page 05	Aluminum T-6
P-TF02	Outer Gear Brace	1	See Page 06	Aluminum T-6
P-TF03	Outer Brace	1	See Page 07	Aluminum T-6
P-TF04	Base Plate	1	See Page 08	Aluminum T-6
P-TF06	Gear Spacing Block	6	See Page 09	Aluminum T-6
Tensile Stage Grip Parts:				
P-SG01	Specimen Grip Clamp	2	See Page 11	SS 304
P-SG02	Right Specimen Grip	1	See Page 12	SS 304
P-SG03	Left Specimen Grip	1	See Page 13	SS 304
P-SG04	Load Cell Grip	1	See Page 14	SS 304
P-SG05	Specimen Pin	2	See Page 11	SS 304
Tensile Stage Power Train and Misc. Parts:				
P-TF05	Grip Power Screw	2	See Page 10	Hardened Steel
P-TF07	Input Shaft Adapter	1	See Page 10	Brass
P-TF08	Input Drive Shaft	1	See Page 09	Hardened Steel
P-TF09	Long Gear Shaft	1	See Page 09	Hardened Steel
P-TF10	Short Gear Shaft	1	See Page 09	Hardened Steel
P-TF11	Step Motor Input Shaft	1	See Page 09	Brass

Micro-Tensile Stage
Assembly View Of
Machined Parts

P-TF11
P-TF05
P-TF03
P-TF04
P-SG05
P-SG01
P-SG03
P-SG04
P-TF01
P-TF06
P-TF02
P-SG02
P-TF08
P-TF07
P-TF09
P-TF10
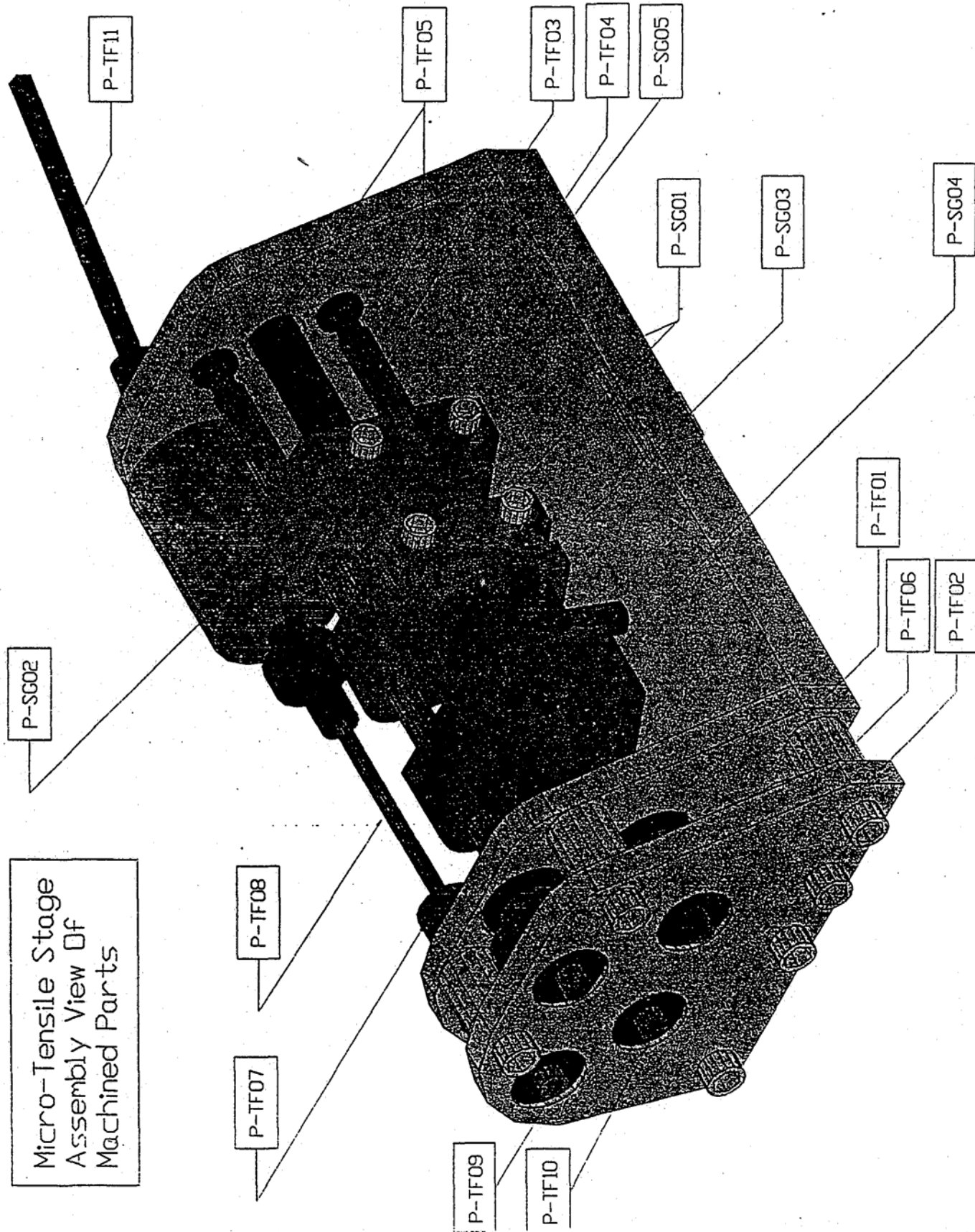
Manufactured Parts Inventory:

Item	Description	Part #	Quant	Price $
Gear One (A)	Spur Gear: Pitch DIA:0.875": Teeth:56 Press.Ang. 20; Quality:10 Page 208, Berg B92 Cat.	Berg P64S28-56	2	10.88 *21.76*
Gear Two (B)	Spur Gear: Pitch DIA:1.25": Teeth:80 Press.Ang. 20; Quality:10 Page 208, Berg B92 Cat.	Berg P64S28-80	1	12.94
Gear Three (C)	Spur Gear: Pitch DIA:0.50": Teeth:32 Press.Ang. 20; Quality:10 Page 208, Berg B92 Cat.	Berg P64S28-32	1	8.90
Slip Clutch (D)	In Line Slip Clutch: Variable Slip Torque to 50in.oz; Pin Hub Style; 1/8" Bore Page 503, Berg B92 Cat	Berg CO16-1-50	1	130.94
Flanged Ball Bearing (E)	ABEC-7 Stainless Steel Bearings; Press In Fit; Bore:0.25"; Chamfer: Page 540, Berg B92 Cat	Berg B2-7	8	14.51 *116.08*
Teflon Bearing (F)	Flanged Press In Fit Bearing; Bore 0.188" Page 546, Berg B92 Cat	Berg B9-6	2	2.22 *4.44*
Speed Reducer (G)	20:1 Speed Reducer;Shaft Rotation "Same" Page 336, Berg B92 Cat	Berg RX11-5	1	370.22
Universal Joint (H)	Stainless Steel Universal Joint; Bore:1/8" Page 495, Berg B92 Cat	Berg UJ-1	1	53.19
Load Cell (L)	100lb Load Cell, Conditioner and associated support devices. "Model 31"	Sensotec AL311	1	
LVDT (J)	LVDT Strain Gage, Conditioner and associated support devices.	RDP Group D-5 Series	1	
Screws				
#4-40 (1)	Set Screw for Part {P-TF07} Length;0.15"		2	Stock
#5-40 (2)	Set Screw for Part {P-SG03} Length: 1/2"		1	Stock
#6-32 (3)	Cap Screw for Speed Reducer Length: 3/8"		3	Stock
#8-32 (4)	Cap Screw for Part {P-SG01} Length: 5/8"		4	Stock
#10-32 (5)	Cap Screw for Part {P-TF01} Length: 1.0"		3	Stock
#10-32 (6)	Cap Screw for Part {P-TF01} Length: 1.5"		3	Stock
#10-32 (7)	Cap Screw for Part {P-TF01} Length: 3/4"		3	Stock

Micro-Tensile Stage
Manufactured Parts
Assembly View

Front View:
Bolt Pattern Detail

Tap for #10-32 screw

2.250"

2.000"

0.250"

Drill for #10-32 screw Three Holes

1.250"

0.333"

1.000"

1.666"

2.775"

3.000"

Tap for #10-32 screw

Tap for #10-32 screw

Plate Thickness: 0.25"

Front View:
Outer Machining Detail

2.500"

2.000"

1.500"

0.500"

1.550"

Rad 0.50"

2.650"

3.000"

(P-TI-01) Inner Gear Brace
Quant (1), Material:(Al,T-6)

Rad 1.00"

0.25"

Rad 0.250"

Front View:
Bearing Insert
Hole Detail

2.0625"

1.719"

1.2115"

1.100"

0.6050" +0.0002" Bore
−0.0000"
Four Holes

1.4635"

2.0625"

2.5290"

3.1480"
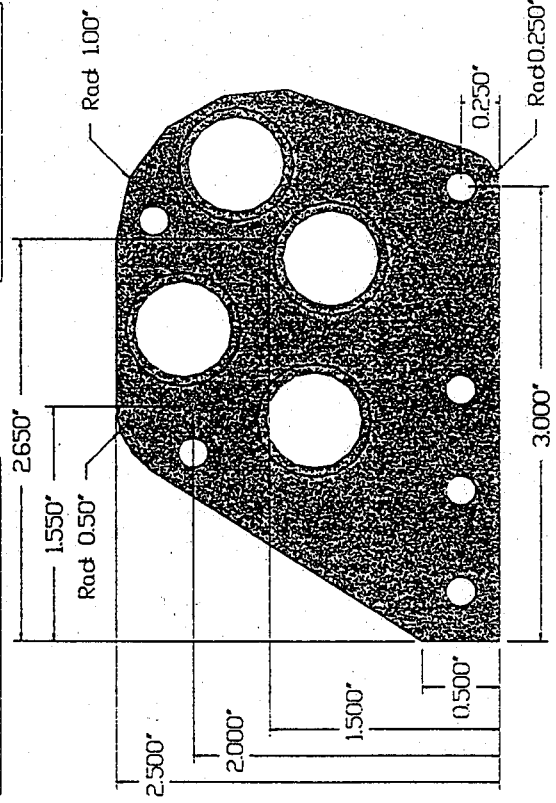
0.75" Bore
Depth: 0.05"
Four Holes

Front View:
Bolt Pattern Detail

Drill for #10-32
screw, 6-Holes

2.775'
1.250'
3.000'
0.333'
1.000'
1.666'
2.000'
2.250'
0.250'

Front View:
Outer Machining Detail

(P-1F02) Outer Gear Brace
Quant (1), Material:(Al,T-6)

Rad 1.00'
Rad 0.50'
Rad 0.250'
0.250'
2.650'
1.550'
3.000'
2.500'
2.000'
1.500'
0.500'

Plate Thickness:0.25"
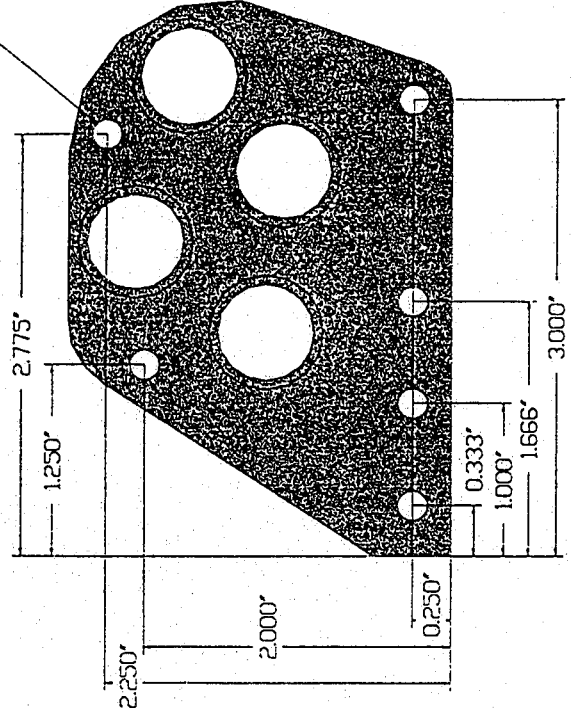
Front View:
Bearing Insert
Hole Detail

0.75' Bore
Depth 0.05'
Four Holes
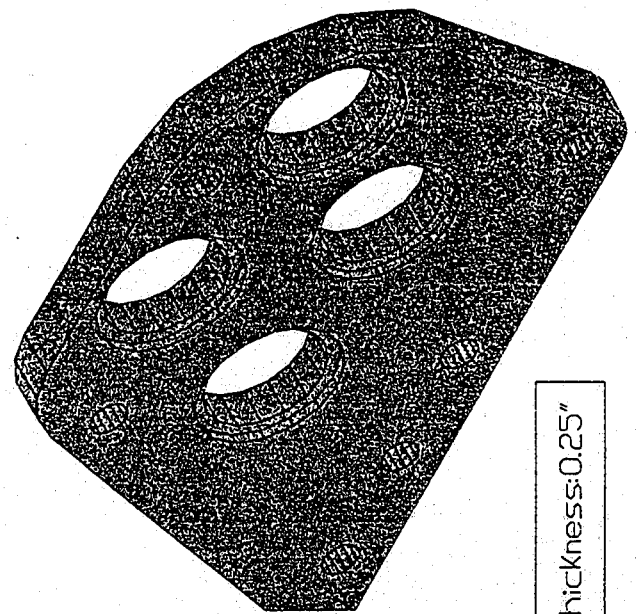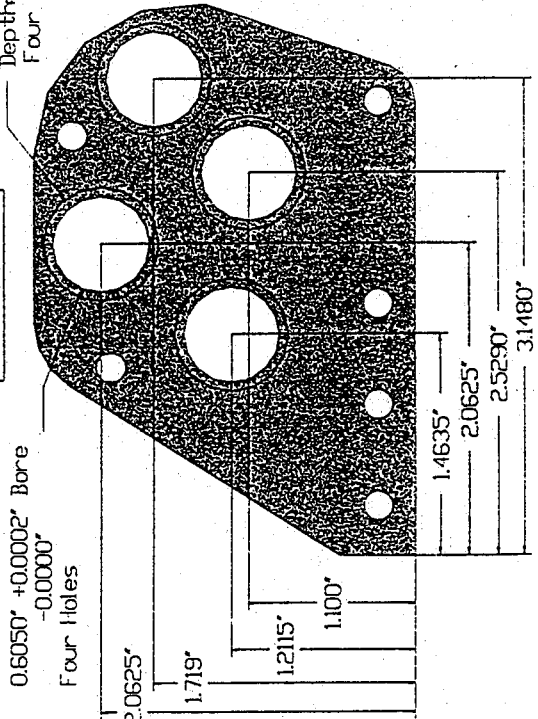
0.6050' +0.0002' Bore
        -0.0000'
Four Holes

2.0625'
1.719'
1.2115'
1.100'
1.4635'
2.0625'
2.5290'
3.1480'

2.0625"

1.2115"

0.500"

0.250"

2.063"

1.464"

0.333"

1.000"

1.667"

3.148"

Bore DIA:0.300"
Depth:0.200"

Bore
DIA:0.525"

Plate Thickness:0.25"

Front View:
Outer Machining Detail

2.500"

2.000"

1.500"

0.500"

1.550"
Rad:0.50"

2.650"

3.000"

Rad:1.00"

0.250"

Rad:0.25"

(P-TF03) Outter Brace
Quant:(1); Material:(Al,T-6)

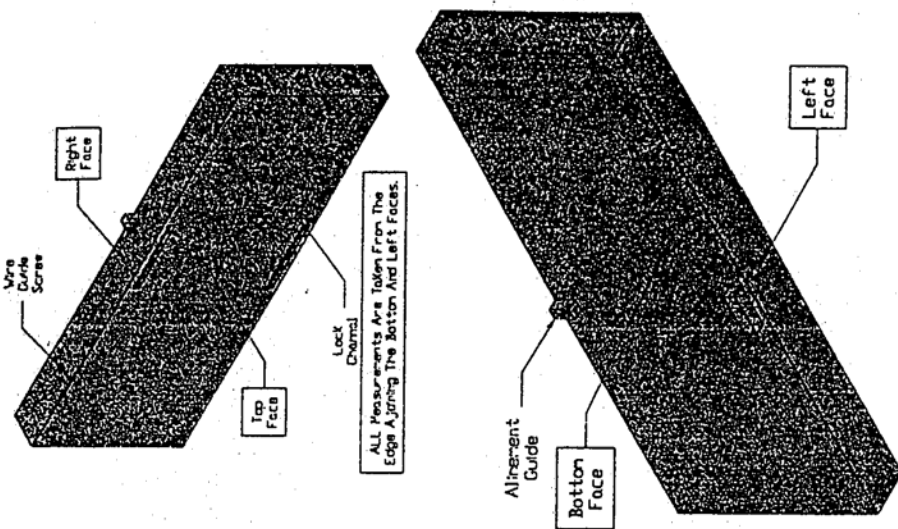Drill and Counter Bore for #6-32
Screw, Three Holes:0.75" Bolt Circle

2.094"

1.532"

2.823"

3.148"

3.473"

1.763"

1.637"
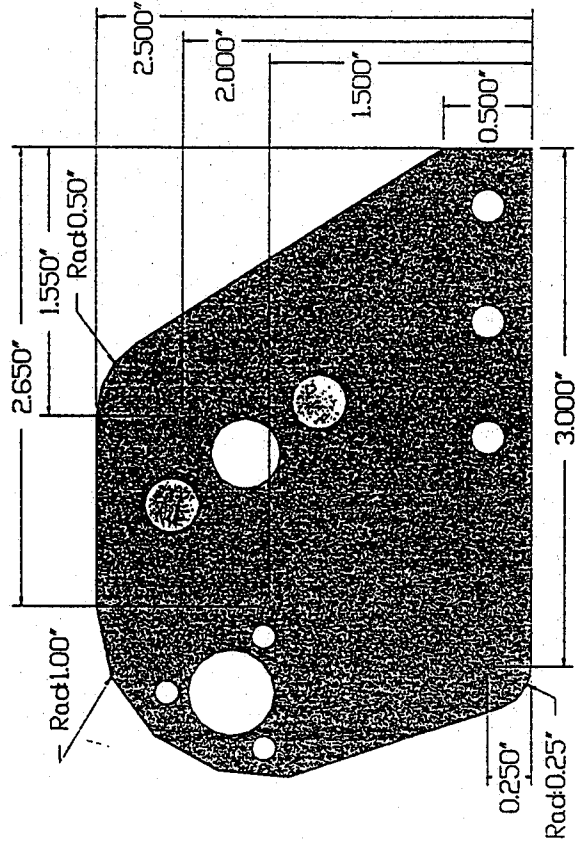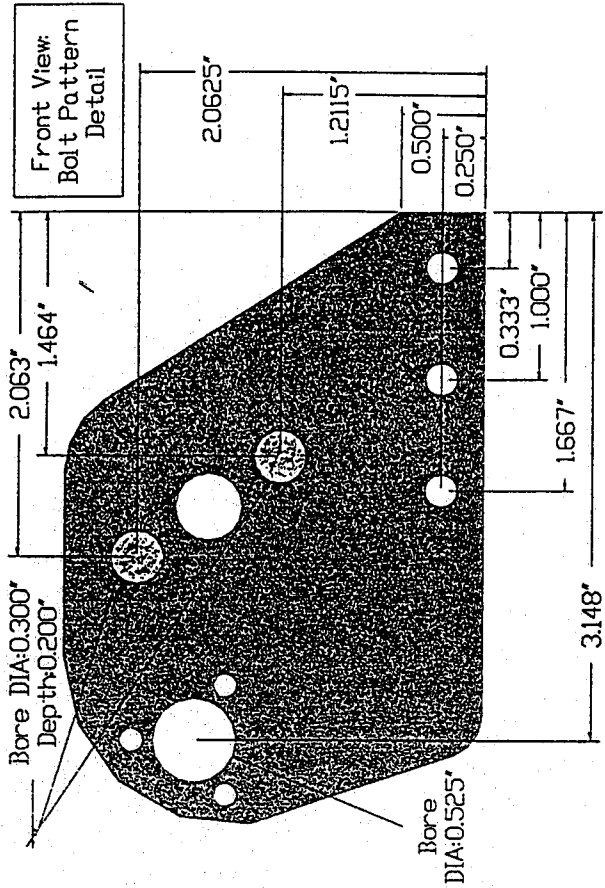
Rear View:
Speed Reducer
Bolt Detail

Right Face

Wire Guide Screw

Top Face

Lock Chanel

ALL Measurements Are Taken From The Edge Joining The Bottom And Left Faces.

Left Face

Alinement Guide

Bottom Face

End View: Bolt Detail, Both Ends Identical

1.666"
1.000"
0.333"
0.250"
0.500"

Tap for #10-32 Screw, Three Holes Depth:0.75"

〈P-TF04〉 Base Plate
Quan't:〈1〉; Material:〈AL,T-6〉

2.560"
2.440"

Alinement Guide

2.275"
2.725"
5.500"

1.850"
0.199"
0.079"
2.050"

Bottom View: Alinement Guide Detail

Top View:Lock Chanal Detail

3.000"
2.000"
1.892"
1.971"
0.079"
2.000"
3.000"

Object Rotated 180 Degrees "End to End" From the Bottom View

Tap for #3-48 Screw, Depth:0.5", Two Holes

1.000"
4.500"

Right End View: Wire Guide Screw Detail

<P-TF06> Gear Spacing Block
Quant <6>; Material:<Al, T-6>

DIA:0.50"

Drill for
#10-32
Screw

Thickness: 0.5625"

Hardened Steel Shaft
DIA: 0.1250"

2.500"

<P-TF08> Input Drive Shaft
Quant:<1>

<P-TF11> Step Motor Input Shaft
Quant: <1>; Material: <Brass>

Shaft 0.1250"
Square

Length 3.00"

Hardened Steel Shaft
DIA: 0.250"
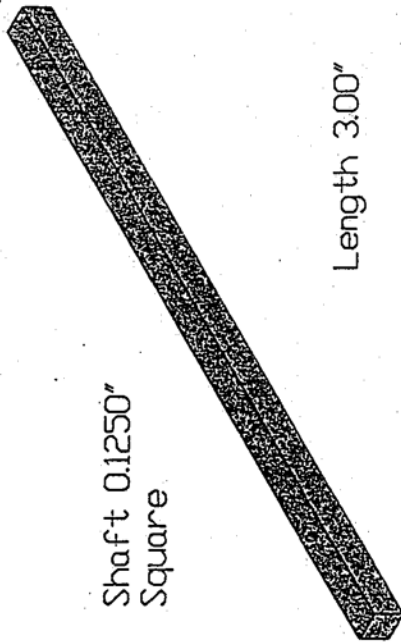
Length

<P-TF09, P-TF10> Gear Shaft
Quant: <1 each>

<P-TF09> Length:1.5625"
<P-TF10> Length:1.0625"
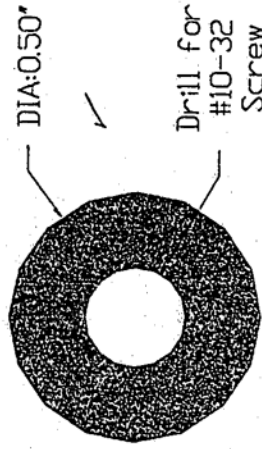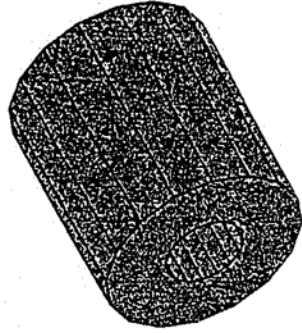
(P-TF05) Grip Power Screw
Quant: (2), Material: (Hardened Steel)

Grid to DIA:0.185"

Right Hand Thread
0.25" - 32

0.25" Nonthreaded Shaft

Left Hand Thread
0.25" - 32

0.50"

2.75"

4.10"

6.875"
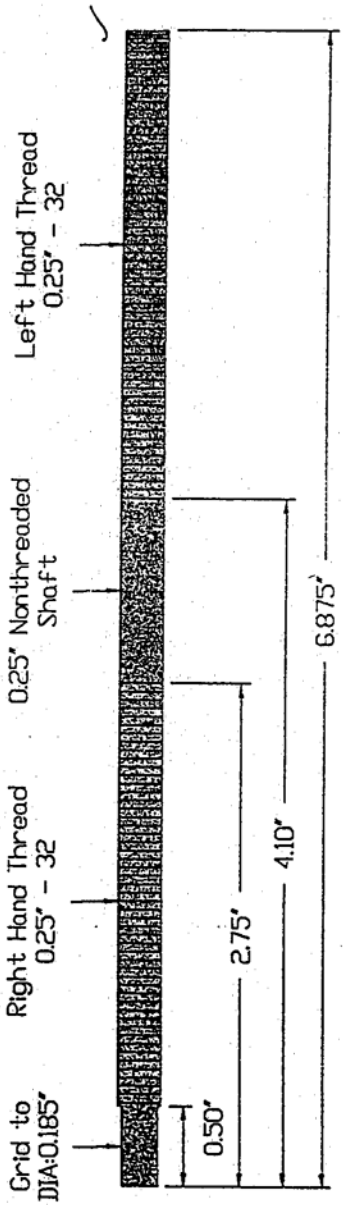
(P-TF07) Input Shaft Adaptor
Quant:(1); Material: (Brass)

DIA:0.50"

Bore DIA:0.1250"
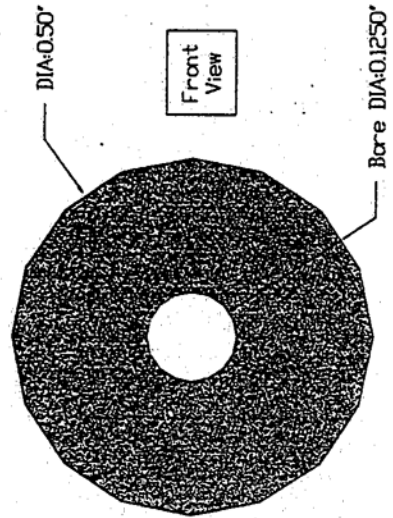
Front View
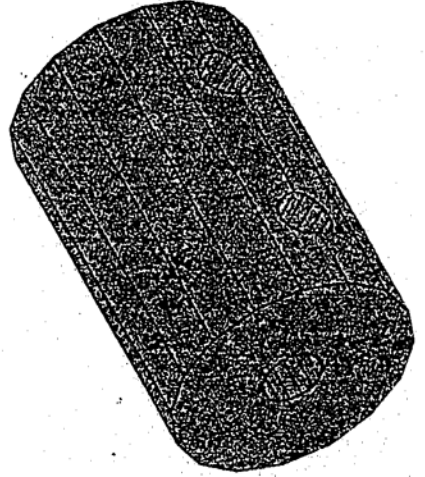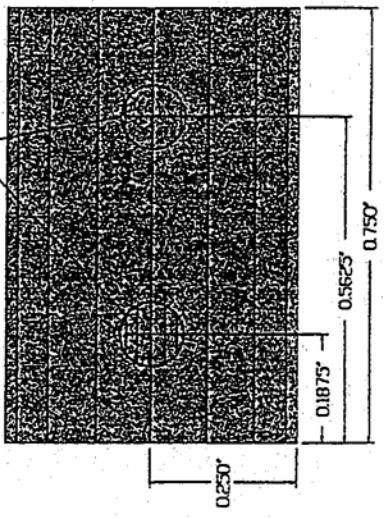
Bore DIA:0.250"
Depth:0.375"

Rear View
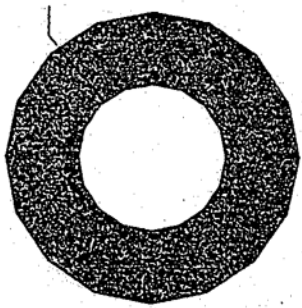
Tap #4-40 for Set Pin
Depth:0.25"

0.750"

0.5625"

0.1875"

0.250"

Right View

[P-SG01] Specimen Grip Clamp
Quant. [2] Material: [SS304]

V-Groved Grip Area
Grove Depth of 1/32"

Drill for #8
Cap Screw

Thickness
of 0.313"

0.0938" Drill
for Set Pin

Drill for #8
Cap Screw
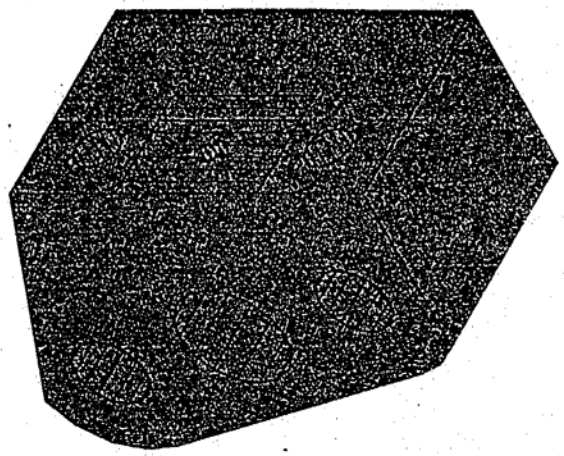
0.219"
0.375"
0.625"
0.875"
1.031"
1.250"

0.375"
0.1875"
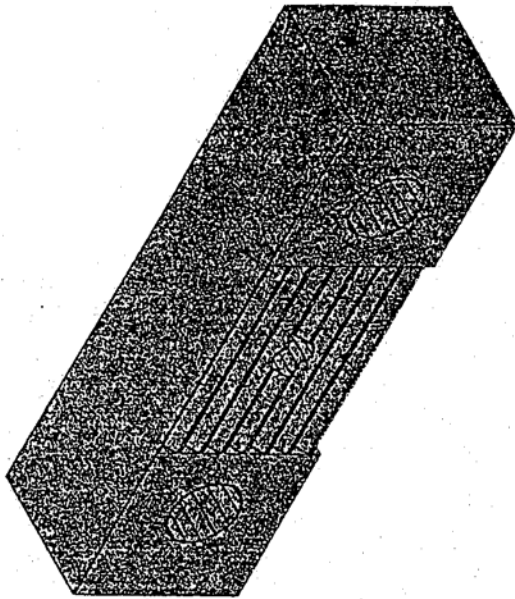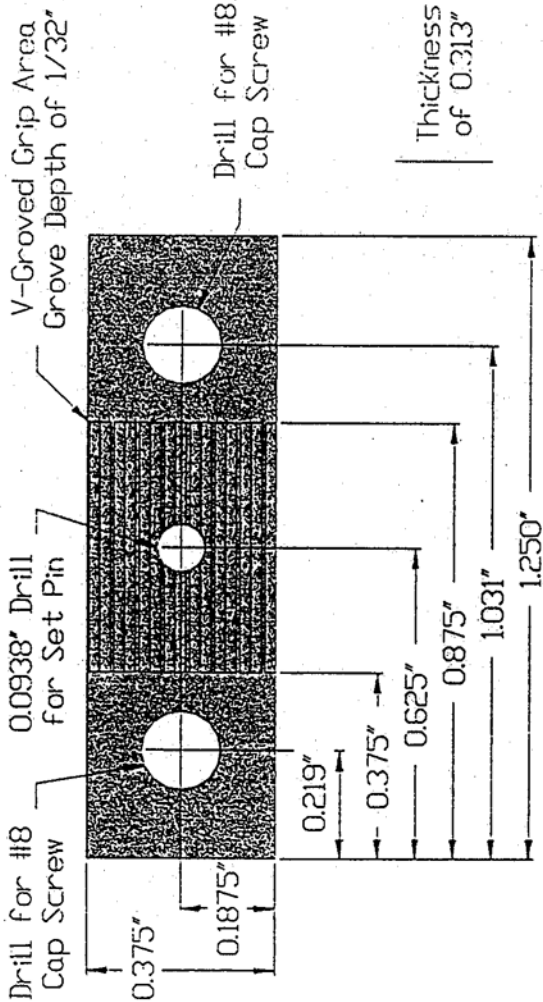
(P-SG05) Specimen Pin
Quant (2); Material:(SS304)
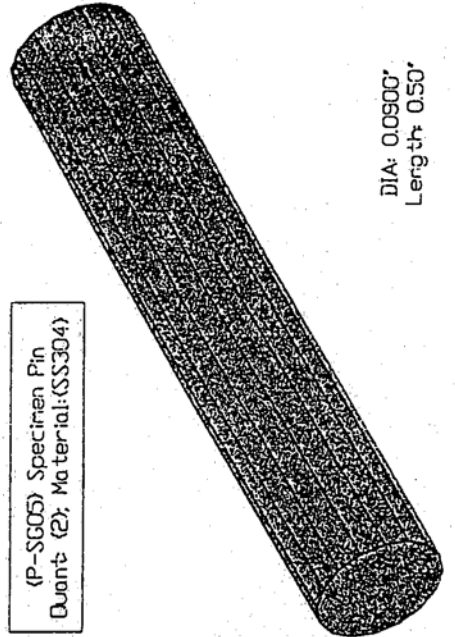
DIA: 0.0900'
Length 0.50'

Top View

(P-SG02) Right Specimen Grip
Quant: (1), Material: (SS304)

Rad0.313"

Right Hand
Thread
0.25" - 32

0.375"-24
Screw Tap with
0.5" Dia. Bore,
Depth of 0.125"

Right Hand
Thread
0.25" - 32

Rad0.313"

0.438"
0.583"
0.727"
0.840"

1.600"

1.250"

0.75"

0.25"

Left
View

#8-32 Tap
Depth:0.312"

V-Grove Grip Area
Depth: 0.0313"

0.0938" Drill
Depth:0.188"

#8-32 Tap
Depth:0.312"

0.1875"
0.375"
0.750"

1.031"
0.875"
0.625"
0.365"
0.219"

Front
View

Rad:0.313"

0.255" Drill

Drill for #4 Screw

0.255" Drill

Rad:0.313"

<P-SG03> Left Specimen Grip
Quant:<1>, Material:<SS304>

1.540"

1.142"

1.250"

0.750"

0.250"

0.750"
0.875"
1.040"
1.156"

Front View

#8-32 Tap Depth:0.312"

V-Grove Grip Area Depth:0.0313"

#8-32 Tap Depth:0.312"

#5-40 Tap See Detail <A>

0.250"

1.031"

0.875"

0.375"

0.219"

0.375"
0.5625"
0.750"

Left Veiw

Fillet Rad:0.0625"

Top View

0.313"

0.75"

0.50"

#10-32 Tap Depth:0.250"

Rear View

0.625"

0.313"

#5-40 Tap for Set Screw

<Detail A> Set Screw

0.700"

Front View

<P-SG04> LC. Grip
Quant:{1}, Material:<SS304>

Rad:0.313"

Left Hand
Thead
0.25"-32

#10-32 Tap
Depth:0.5"

Left Hand
Thread
0.25"-32

Rad:0.313"

1.540"

1.250"

1.142"

0.625"

0.250"

0.313"

0.750"

1.039"

1.156"

Thickness:
0.750"

Power and Gear Train
Assembly View

Step Motor
Input Shaft

U-Joint

Speed Reducer

Slip Clutch

Input Drive
Shaft

Input Shaft
Adapter

Long Gear Shaft

Gear Three

Gear
Two

Short
Gear Shaft

Gear
One

In Blue
4 Ball
Bearings

In Blue
4 Ball
Bearings

Gear
One

Load Cell
Grip

100lb Load
On Specimen

Left Grip

Right Grip

Power Screw
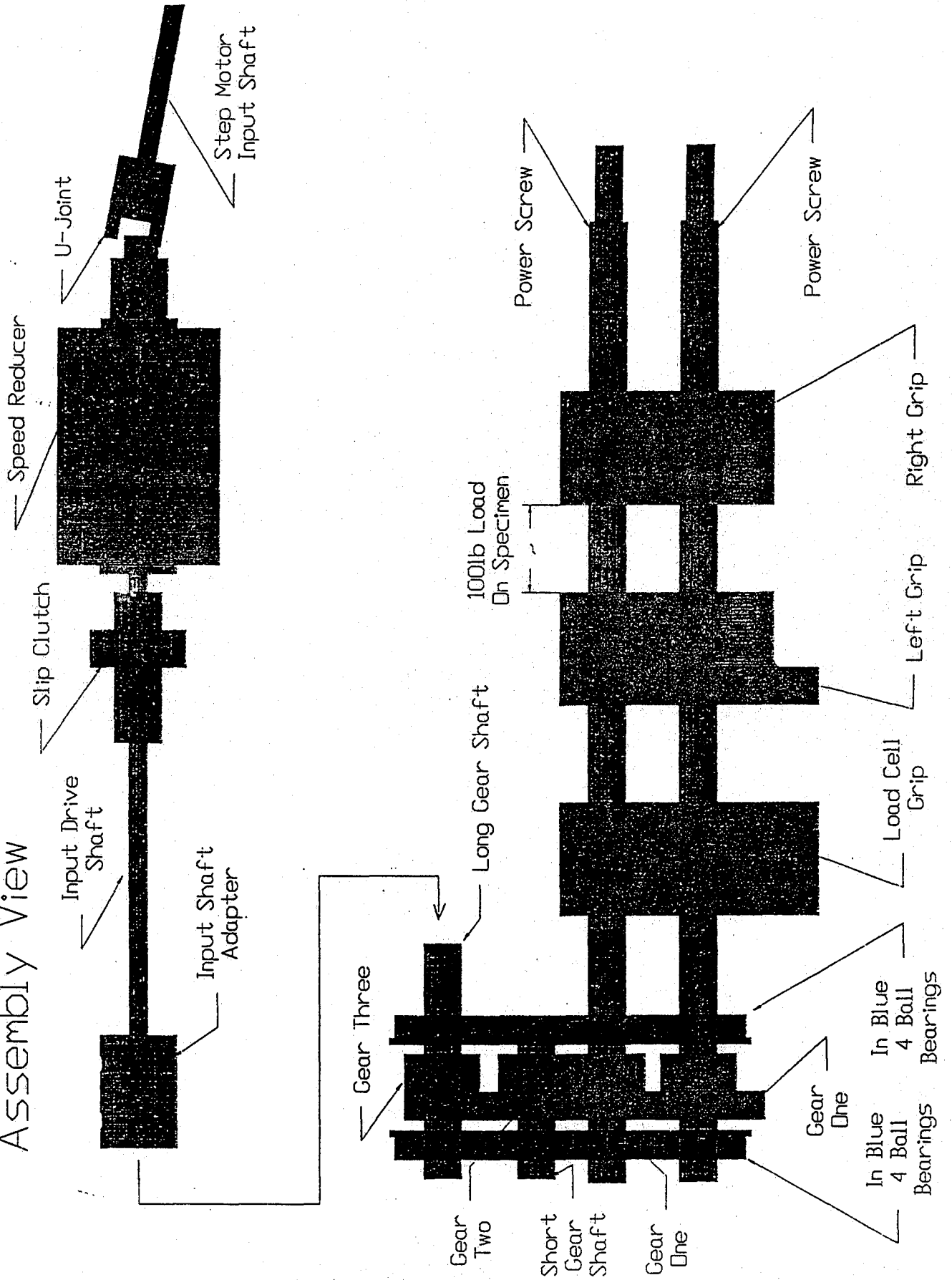
Power Screw

Micro-Tensile Stage Gear Train:
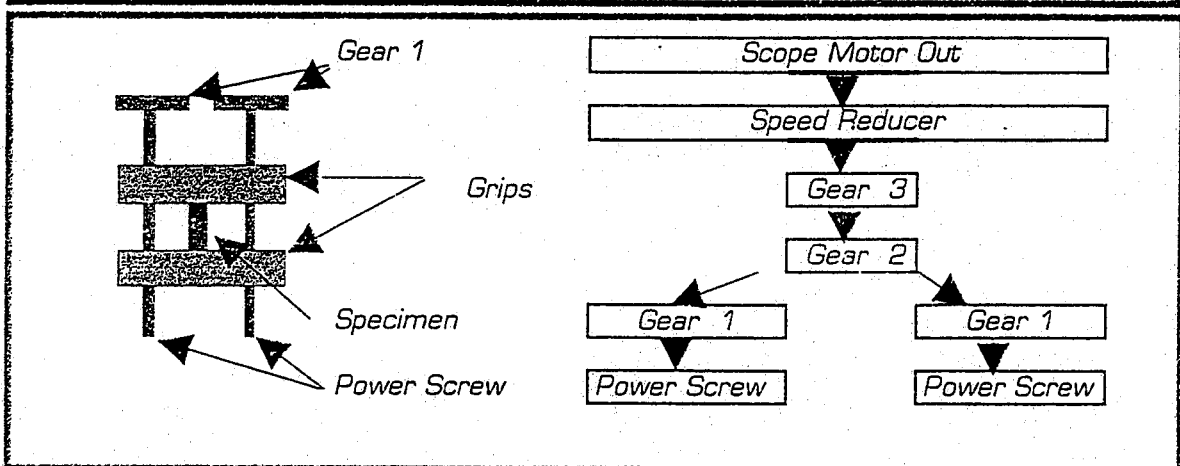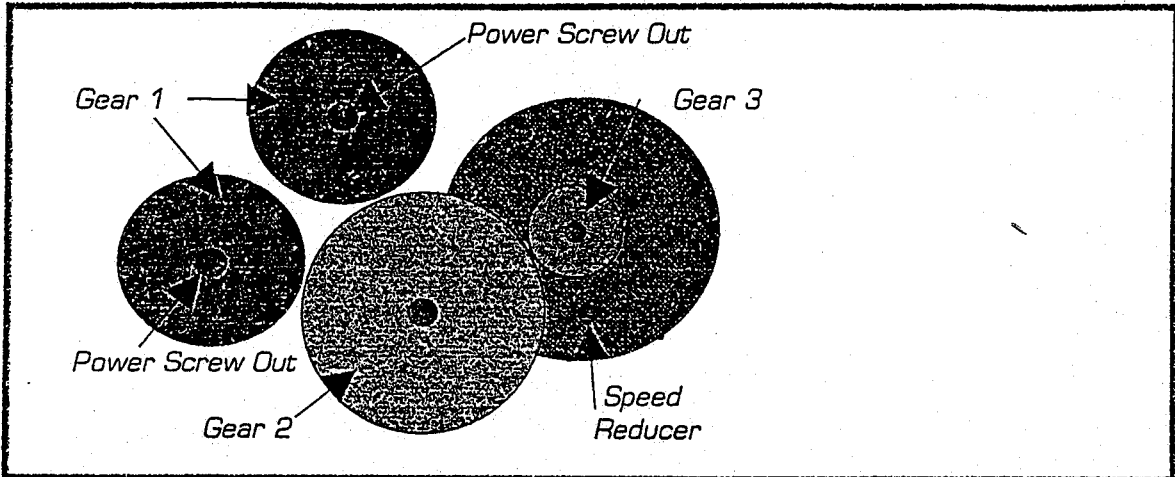
Power Screws:	0.25"- 32
Desired Strain Rate:	10E-5(in/in)/s
Guage Length:	3/8"
Desired Tensile Load Produced:	100lb.

Scope Motor Rotational Rates:
0.1312 rpm
0.2625 rpm
0.5249 rpm
1.0499 rpm
2.0997 rpm
4.1995 rpm

Gear 1:P64S28-56
Teeth: 56
Pitch Dia. 0.8750"
O.D. 0.906"
Gear 2:P64S28-80
Teeth: 80
Pitch Dia. 1.25"
O.D. 1.281"
Gear 3:P64S28-32
Teeth: 32
Pitch Dia. 0.5"
O.D. 0.531"
Gear Qual. 10
Press. Angle   20
Stainless Steel 303

Rotational Velocity

Power Screws

Screw	0.25" DIA. 32 turns/in
Strain Rate	1.00E-04 (in/in)/s
Gauge length	0.375 in
0.5 Grip Velocity	1.88E-05 in/s

0.5 because both grips are moving and we are interested in power screw velocity required to make one grip move half of the velocity.

Power Screw Rotation = 6.00E-04 rev/s
0.036 rpm

Rotation required to produce desired strain rate.

Gears

Gear 1 = 0.036 rpm *Rotation of Gear One.*

$w1*r1=w2*r2$
radius of gear one	0.4375 in
radius of gear two	0.625 in

Gear 2 = 0.0252 rpm *Rotation of Gear Two.*

$w2*r2=w3*r3$
radius of gear three	0.25 in

Gear 3 = 0.063 rpm *Rotation of Gear Three.*

Speed Reduction

Step Motor Input   0.131234 rpm

SR=Input/Output = 2.083079 rpm *Speed Reduction at lowest input possible.*

Torque Analysis:

Torque on power screw $T=(Fd/2)(l+pi*u*d)/(pi*d-u*l)$

F=	50 lb
d=	0.229703 in
u=	0.05 assumed friction
l=	0.03125 in

Torque= 0.536978 lb in
8.591645 oz in

Torque require to produce a loading of 100lb in specimen.

Manufactured Parts

Gear One	Cat. #	Torque Max		Price per	Sub-total
1/4 bore 3/16 Face	P64S28-56			10.88	21.76
		8.16in oz			
1/4 bore 1/4 face	P48U66-42			13.51	27.02
Gear Two					
1/4 bore 3/16 face	P64S28-80			12.94	12.94
		24.5in oz			
1/4 bore 1/4 face	P48U66-60			15.88	15.88
Gear Three					
1/4 bore 3/16 face	P64S28-32			8.39	8.39
		3.8in oz			
1/4 bore 1/4 face	P48U66-24			11.39	11.39

Slip Clutch	CO16-1-50	Max Torque 50in oz		3.67	20.74
Ball Bearing	B2-7	DIA for fit DIA 0.2		14.51	116.08
Teflon Bearing	B9-6	DIA for fit DIA 0.2		2.22	4.44
Speed Reducer	RX11-5	Torque Out 65ozin		370.22	370.22
U-Joint	UL-1			53.19	53.19
			Total $		587.53