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FORMATION OF EPOXY COMPOSITES FROM SINGLE  
SHORT FIBERS

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Monsanto Research Corporation

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Starting with very low concentrations ( $v_f > 0.01$ ), single short fibers can be unidirectionally oriented, statistically overlapped and effectively packed into highly filled, shaped granules or impregnated tapes with concentrations as high as  $v_f = 0.6$ . This packaging of fibers was demonstrated using E-glass, graphite and boron fibers and a binder of epoxy resin.

The mechanical properties of the composites formed by molding the granules and tapes of fibers are superior to those reported previously. In particular, the strength utilization factor for E-glass fibers and epoxy resin composites is increased from the normal level (0.25 to 0.55) to a higher level (0.55 to 0.88).

The main reason for this increase is the statistical distribution of single fibers in the composites which requires a much higher percentage of fiber breakage when the composite fails under stress. Fiber orientation, damage, wetting, void content and the adhesion with resin are also limiting factors on the strength utilization factor.

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by

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FOREWORD

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FORMATION OF EPOXY COMPOSITES FROM SINGLE SHORT FIBERS

by

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INTRODUCTION

There are a number of methods to incorporate short reinforcing fibers into composites with thermoset resins.<sup>1-7</sup> In all of the methods, the major goal is good fiber distribution, controlled orientation, wetting and adhesion of fibers with resin, and a high fiber content. Superior mechanical properties of reinforcing fibers cannot fully be utilized in a composite unless the fibers are properly incorporated. Both the modulus and strength utilization factors, defined as the respective modulus and strength ratios of the discontinuous to the continuous fiber composites, are functions of the fiber content, the modulus or strength ratio of the fiber to the resin, the aspect ratio of the fiber,<sup>6,8</sup> the extent of fiber orientation,<sup>9,10</sup> the void content<sup>7,11</sup> and the adhesion of resin with fiber surfaces.<sup>7</sup> The modulus utilization factors observed<sup>2-7,9,10</sup> for 1/16" to 1/2" long glass, boron and graphite fibers are in the range of 0.18 to 1.00. On the other hand, the strength utilization factors observed are usually between 0.25 and 0.55,<sup>2-7,9,10</sup> with the upper limit being 0.75.<sup>6,7</sup>

It is the purpose of this report to demonstrate that the information<sup>12</sup> available on fiber behavior in dilute suspensions

can be utilized for the preparation of composites from unidirectionally oriented and statistically overlapped single short fibers, and further, that the composites prepared by this method show high strength utilization factors, in the range of 0.54 to 0.88. For the formation of composites reported here, short fibers are first individually dispersed and unidirectionally oriented in dilute suspensions subjected to steady shear flow. The suspending medium is then removed without disturbing the fiber orientation. The resulting fiber mat, in which fibers are statistically overlapped and unidirectionally oriented, is impregnated with resin so that the resulting fiber concentration,  $v_f$ , is 0.3 to 0.6. From this resin-impregnated mat, granules or tapes of short fibers are prepared. The granules can be extruded into strands if necessary. Composites are compression molded from the granules, tapes or extruded strands. The effectiveness of this fabrication process is examined in terms of the extent of fiber orientation, overlapping, and damage, wetting of fibers with resin, and the mechanical properties of the composites. E-glass, Thornel 25 graphite and boron fibers were used in the experiments. Flexural modulus and strength were measured.

### EXPERIMENTS AND RESULTS

Preparation of granules and tapes: Filers in the form of chopped strands were first dispersed into single fibers using glycerol as a suspending medium, at very low concentrations (0.1 to 0.75 vol.%). The fibers used are listed in Table I. The short fibers are easily debundled and dispersed into individuals in a viscous medium after the treatment described in the table.

The fibers were then unidirectionally oriented in a modified Couette apparatus as shown in Figure 1. The radii of the rotating disk and the sieve with a 140 mesh screen are 3" and 4", respectively. The central part of the screen just beneath the rotating disk is covered by a rubber disk to protect fibers from the high shear arising from the narrow clearance. The rotational speed of the disk is variable from 0 to 150 rpm. The axes of the disk and the sieve are matched by using a tight-fitting lid. After the fibers were unidirectionally oriented, glycerol was drained off from the bottom of the apparatus. A reduced pressure facilitates the filtration of the viscous fiber suspension.

The resulting fiber mat was washed with hot (60 - 65°C) water and acetone. The fiber mat was then immersed in an acetone solution of partially cured resin, coupling and wetting agents. The resin system used was Epon Resin 828 (Shell Chem. Co.)

cured with 4,4'-methylenedianiline (Eastman Org. Chem.), 25 phr, at 80°C for 10 minutes. A silane coupling agent (A-1100, Union Carbide Co.) and a wetting agent (Arquad 12-50, dodecyl trimethyl ammonium chloride, 50% active, Armour Ind. Chem. Co.) were used for glass and boron fibers. A cationic wetting agent (Triton X-400, 25% active, Rohm and Haas) was used for graphite fibers. The acetone was then evaporated at 80°C in a vacuum oven for 20 minutes. The rest of the acetone which remained was evaporated at room temperature. The resin-impregnated mat consisted of unidirectionally oriented and statistically overlapped fibers.

Pointed granules of resin-coated fibers could be produced by dispersing the impregnated fiber mat in hot water agitated by a propeller stirrer, followed by cooling of the aqueous dispersion to 0°C. However, nonuniform distribution of resin on fiber surfaces occurred on some granules.

As an alternative for achieving a more uniform distribution of resin on fiber surfaces, the impregnated fiber mat is dispersed in glycerol a second time instead of the hot water. The resin-coated fibers in the mat were dispersed into individuals and oriented in the Couette apparatus. Glycerol was drained off and the resulting impregnated tape of short fibers was washed with cold water without disturbing the fiber orientation. Although a noticeable amount of resin was removed from the fiber surfaces in this process, uniform distribution

of resin on fiber surfaces could be obtained. To minimize the removal of resin, the time that the dispersion was subjected to shear in glycerol was kept at a minimum. In the impregnated tape, fibers are individually coated with resin, unidirectionally oriented and statistically overlapped. When the impregnated tape was dispersed in hot water, agitated by a propeller stirrer, and the temperature of the dispersion cooled to 0°C, pointed granules of uniformly resin-coated fibers were obtained. The size and shape of the granules are functions of resin content, temperature of the aqueous dispersion, agitating speed, time, cooling speed and fiber length.

The granules made from 1/8" long glass, graphite and boron fibers are shown in Figure 2. The shape of granules becomes more pointed when they are made at higher shear rates. The length of granules increases as the length of fibers increases. Resin-impregnated tapes could be prepared for glass and graphite fibers. However, boron fibers could not be impregnated in the form of tapes due to the rapid sedimentation in glycerol. Again the granules and tapes consist of fibers individually coated with resin, unidirectionally oriented and statistically overlapped. They are in a form which can be processed directly by compression molding.

Formation of composites from granules and tapes: The granules of impregnated short fibers prepared by the preceding processes were compression molded using the following procedure.

The granules were carefully placed in the mold using a pair of tweezers, such that they were well overlapped and unidirectionally oriented in a mold with typical dimensions of 1/4" x 4" by 1/16" in depth. The tapes of impregnated short fibers were cut to fit the mold. The resin and hardener mixed in the same proportions as before were added to the granules and tapes prior to the molding of the composites. Composites were molded by using a laboratory press under the following conditions: 15 minutes at 105°C and 100 psi, 10 minutes at 190°C and 150 psi, and 30 minutes at 190°C and 1,500 to 2,500 psi. The composites molded were post-cured at 150°C for a few hours.

Measurements of mechanical properties: The flexural strength,  $\sigma$ , and flexural modulus,  $E$ , of the composites were measured at three different positions on each specimen. A constant crosshead speed, 0.02"/min., was used for 1" span tests on a table model Instron tester at room temperature,  $24 \pm 1^\circ\text{C}$ . The results are listed in Tables II and III, and are also compared with data reported<sup>2</sup> in the literature with the same fibers and matrix. The results are also shown in Figures 3, 4 and 5.

The utilization factors of fibers are calculated from the ratios of the measured to the calculated values of strength,  $\sigma$ , and modulus,  $E$ , of composites as follows:

for strength:  $f_\sigma = \sigma/\sigma_c$

for modulus:  $f_E = E/E_c$

where

$$\sigma_c = v_f \sigma_f + (1 - v_f) \sigma_r \quad (1a)$$

$$E_c = v_f E_f + (1 - v_f) E_r \quad (1b)$$

and c, f and r refer to composite, fiber and resin, respectively. The values of  $E_f$  and  $\sigma_f$  are listed in Table I. The fiber content,  $v_f$ , was determined by burning the resin from glass and boron fiber composites. The graphite fiber content was determined by  $H_2O_2$  decomposition of resin.<sup>14</sup> The density,  $\rho$ , of composites was measured from the total weight and dimensions of the specimen, and also calculated from the fiber content.

#### COMPOSITE ANALYSES AND DISCUSSION

The composites formed from glass fiber granules, except for a few specimens, demonstrate higher levels in the flexural strength and modulus compared to those obtained from bundled fibers by the encapsulation/extrusion/compression molding technique<sup>2</sup> as shown in Figure 3. The upper straight lines are calculated from the law of mixtures, Eqs. 1a and 1b, for the unidirectional, continuous fiber composites. The strength utilization factor of fibers in these composites is between 0.55 and 0.88. The strength utilization factors observed previously were usually between 0.25 and 0.55<sup>2-7,9,10</sup> although

higher values have been reported in the vicinity of 0.75.<sup>6,7</sup> To identify the key factors which make these composites superior to others, the following structural parameters are examined with the composites: a) fiber damage, b) void content, c) wetting and adhesion, d) fiber orientation, e) type of failure and f) fiber characteristics.

a) Fiber damage: Fiber damage in the composites was measured on the microphotographs of the fibers obtained by burning the resin for determination of fiber content. Fiber damage in the composites made from hand laid-up granules was slightly less than that observed for the composites made by the extrusion/compression molding technique.<sup>2,9</sup> About 20% of the 1/8" fibers were longer than 1/10" in the former composites compared to 70% in the latter at 40 to 60 vol. % fiber content. It is unlikely that this slight change in fiber damage affects the strength level to account for the large difference in the strength utilization factor.

b) Voids: Voids can be visually observed on specimens, and microscopically viewed on the polished cross-sections. Voids are very few in the composites with the highest strength level, but this is not always true for specimens with the highest mechanical properties. The ratio of the measured to the calculated densities listed in Tables II and III serves as a measure of void content.

c) Wetting and adhesion: Wetting and adhesion of resin to fiber surfaces can be observed on electron scanning micrographs of fractured surfaces. Although interfacial failure<sup>10</sup> was also locally observed, the adhesion of resin to fiber surfaces was reasonably good. When the fiber surfaces are well-wetted with resin and the void content is low, the specimen is generally transparent.

d) Orientation: Fiber orientation distributions in typical composite specimens prepared in different ways were measured with respect to the angles,  $\phi'_z$  and  $\theta'_z$ , defined in Figure 6, and shown in Figure 7. The angles  $\phi'_z$  and  $\theta'_z$  were measured, respectively, from the orientation and the length,  $l' = d_f/\sin\theta'_z$ , of the major axis of the elliptical fiber cross-sections at 100 different positions in a polished x-y plane. In Figure 7, the cumulative distribution curves of fiber orientations are plotted against the angles  $\phi'_z$  and  $\theta'_z$ . It is apparent from the comparison of the distribution curves that the fiber orientation in composites made from hand laying granules is not perfect, but superior to those obtained from tapes and from bundled fibers by the extrusion/compression molding technique.<sup>2,9</sup> The higher levels of flexural strength and modulus are, at least, partially due to the better alignment of fibers in these composites. The modulus utilization factor,<sup>8</sup> theoretically predicted for unidirectionally oriented composites of these short E-glass fibers/epoxy systems, is in the range of 0.93 to 1.00. The lower level observed in the modulus of the composites is partially

due to the misalignment of fibers in the composites. It has been theoretically and experimentally known that the modulus of fibrous composites rapidly decreases when the angle,  $\theta_x = \cos^{-1}(\cos\phi'_z \cos\theta'_z)$ , between the fiber axis and the stress axis is around 10 to 30 degrees.<sup>10</sup> The composite strength, on the other hand, rapidly decreases when the angle  $\theta_x$  is between 0 and 20 degrees.<sup>10,15</sup> The fiber alignment in the composites made from impregnated tapes is comparable to those of the composites<sup>9</sup> made by the extrusion/compression molding technique as shown in Figure 7. The moduli observed for the former composites are comparable to those of the latter ones with a few exceptions. However, the strength utilization factors observed for the composites made from impregnated tapes are in all cases in the range of 0.54 to 0.87. As an extreme case, flexural properties of random composites formed from single short fibers are also measured and shown in Table II. In their preparation, 1/8" E-glass fibers were individually dispersed in glycerol, formed into mats without any alignment of the fibers, impregnated with resin and compression molded into a 4" x 4" x 1/16" composite sheet. Test specimens of 1/4" x 1 1/2" x 1/16" were cut out from the sheet in various directions and used for flexural tests. The modulus utilization factor theoretically predicted<sup>16</sup> for random composites is in the range of 0.28 to 0.33. The experimentally observed values of modulus utilization factors are generally in the range of 0.46

to 0.50.<sup>9</sup> The present data support the empirical prediction. The strength utilization factors observed for these random composites are still in the range of 0.46 to 0.64 despite the random orientation of fibers. These results with the composites made from impregnated tapes, and the random composites, indicate that the unidirectional alignment of fibers is not the essential key factor for the high strength utilization factor of short fiber composites.

e) Type of failure: The most significant difference observed between good and poor specimens is in the mode of failure. The mode of failure significantly depends on the fiber distribution in the composites. In the granules of impregnated fibers, fibers are individually coated with resin, unidirectionally oriented and statistically overlapped. Since the composites are compression molded from these granules in the presence of newly added resin and hardener, it is very likely that there is a resin-rich boundary layer between the granules. The thickness of the resin layer depends on the shape of the granules and the total fiber content in the composite. Similar resin-rich granular boundary can be seen<sup>10</sup> in the composites made by the encapsulation/extrusion/compression molding technique. In addition to this, many fibers are still in the original bundle in the latter composites. In the specimens which yield lower strength levels and the composites made from bundled fibers, flexural failure occurs through the

resin-rich boundary region between granules. Such a kind of failure is illustrated in Figure 8a. The fractured surface was dyed with black ink. When the fiber granules are thin and long, as shown in Figure 2a, and well overlapped, the fracture surface demonstrates a jagged path as shown in Figure 8b. In the same specimen, failure can also occur across fiber granules, as illustrated in Figure 8c. When the fibers are as long as 1/4" to 1/2", the length of their granules are 1" to 3" and the failure of the composites always occurs across fibers in the same manner as shown in Figure 8c. High strength levels and high strength utilization factors are observed for these composites as shown in Table II. In the composites made from impregnated tapes and in the random composites, the flexural failure always occurs across fibers, as demonstrated in Figure 8d, since fibers are statistically distributed without forming any granular structure in these composites. This interprets the high level in strengths of these composites.

The key factor to achieve high strength utilization factors is the statistical distribution of single fibers in the composites. The strength of composites is increased by an increase in the surface area generated during failure and by an increase in the number of fibers broken at fracture.

f) Fiber characteristics: Fiber alignment, damage, wetting, void content and the adhesion with resin also become

limiting factors on the strength utilization factor, depending on fiber characteristics. The resin distribution in graphite fiber composites made from the impregnated tapes is less uniform compared to those made from granules. The difficulties in uniform wetting of fiber surfaces with resin and in eliminating voids in the composites result in the poor mechanical properties and reliabilities of these composites. Even with the composites made from granules, the observed highest value in the modulus utilization factor is only 0.56 compared to the range of 0.95 to 1.00 theoretically predicted.<sup>8</sup> This low modulus level may be due to the misalignment of fibers in the composites and the anisotropic nature of the graphite fibers. Since the graphite fiber granules obtained were mostly as short as 1/3" to 1/2" as shown in Figure 2b, the alignment of the granules by hand was very difficult. The failure of graphite fiber composites always occurred along the boundary layer between granules. Consequently, the strength utilization factors observed are in the range of 0.57 to 0.17, as shown in Table III. As an alternative technique for fiber alignment, the granules were also extruded through a 1/8" diameter orifice into a strand and then compression molded. However, flexural strength appreciably decreased in the composites made by this technique, while the modulus did not. The decrease in the strength level is due to the serious damage of fibers in the extrusion process. About 80% of fibers are shorter than 1/26" after the extrusion.

Composites formed from boron fiber granules were not of comparable quality to the composites formed by the hand lay-up technique<sup>6</sup> ( $f_E = 0.48$  to  $0.67$ ) and by the extrusion/compression molding technique<sup>2</sup> ( $f_E = 0.58$ ). The modulus utilization factor theoretically predicted for this system is in the range of  $0.82$  to  $0.86$ .<sup>8</sup> It is apparent from the drastic decrease in the density of the composites, as shown in Table II, that the composites have a high void content. The granules themselves have a large amount of cavities presumably due to the surface roughness of the fibers.<sup>17</sup> The increase in molding pressure was not effective to improve the mechanical properties. Both the strength and modulus utilization factors of boron fiber composites decrease proportionally to the void content. Void content has to be reduced to achieve higher mechanical properties in boron fiber composites.

## CONCLUSIONS

Starting with very low concentrations, we can unidirectionally orient, statistically overlap and effectively pack single short fibers into highly filled granules or impregnated tapes.

The mechanical properties of composites directly compression molded from the granules and tapes of E-glass fibers are superior to those reported previously. In particular, the strength utilization factor can be increased from the prior level of 0.25 to 0.55 reported in the literature to a higher level (0.55 to 0.88). The main reason for this improvement is the statistical distribution of single fibers in the composites which results in effective fiber breakage when the composite is stressed to failure. Fiber orientation, damage, wetting, void content and the adhesion with resin are also limiting factors on the strength utilization factor.

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Table I. Characteristics of fibers and resin.

Material:	Length: (inch)	Diameter: (mil)	Density: (g/cc)	$E_f \times 10^{-6}$ : (psi)	$\sigma_f \times 10^{-3}$ : (psi)	Fiber treatment
E-glass fibers,	1/8	0.6	2.54	9.9 <sup>a</sup>	249 <sup>a</sup>	Heat- treated at 360°C for 12 to 24 hrs
	1/4	0.6				
	1/2	0.6				
Thornel 25 graphite fibers,	1/8	0.3	1.42	24.3 <sup>b</sup>	180 <sup>b</sup>	Washed in hot water at 90°C for 10 minutes
Boron fibers,	1/8	1.3	4.80	60 <sup>c</sup>	318 <sup>c</sup>	Washed with acetone
Epoxy resin <sup>d</sup>	-	-	1.17	0.25	15.4	

a) measured.

b) references 7 and 13.

c) reference 6.

d) Epon Resin 878 cured with 4,4'-methylenedianiline, 25 phr.

Table II. Strength and modulus utilization factors observed for glass and boron fiber composites.

Fiber length: (inch)	Fiber content: (vol. %)	$\left(\frac{\rho_{\text{meas.}}}{\rho_{\text{calc.}}}\right)$	$f_{\sigma}$ (%)	$f_E$ (%)	Type of failure: to (Ref. Figure 8)
a) Uniaxial glass fiber composites made from granules:					
1/8	0	1.00	-	-	-
	20.9	0.97	70	66	B
	35.6	0.94	77	83	B
	40.4	0.91	84	82	B
	49.0	1.02	58	96	A
	54.7	1.00	55	75	A
	55.5	1.04	82	104	B
	61.3	0.98	77	100	B
1/4	56.9	1.04	85	62	C
1/2	57.0	1.08	88	64	C
b) Uniaxial glass fiber composites made from tapes:					
1/8	38.0	0.99	67	38	D
	40.8	1.02	87	80	D
	45.5	1.02	54	69	D
	52.4	1.03	62	35	D
	56.0	1.00	61	86	D
	1/4	20.0	0.98	81	68
1/2	58.6	0.93	58	48	D
	49.1	1.03	65	86	D
c) Random glass fiber composites:					
1/8	15.9	-	64	51	D
	19.2	-	58	48	D
	22.6	-	54	49	D
	18.7*	-	46	48	D
	20.7*	-	50	50	D
	23.6*	-	54	50	D
	25.5*	-	49	46	D
d) Uniaxial boron fiber composites:					
1/8	38.9	0.69	29	19	A
	40.2	0.64	22	15	A
	40.4	0.64	10	9	A
	51.5	0.61	9	6	A

\*Flexural properties were measured in the transverse direction.

Table III. Strength and modulus utilization factors observed for 1/8" graphite fiber/epoxy composites.

Fabrication method:	Fiber content: $\left(\frac{\rho_{\text{mean.}}}{\rho_{\text{calc.}}}\right)$ :	$f_{\sigma}$ :	$f_E$ :	
	(vol. %)	(%)	(%)	
From granules	16.4	0.97	63	32
	19.0	0.96	66	38
	19.6	1.00	65	37
	20.4	0.98	64	42
	26.5	1.04	71	56
	28.7	1.02	60	30
	29.7	1.01	57	37
	29.7	0.96	59	35
	32.8	0.98	60	47
From tapes	36.6	1.00	65	30
	41.5	1.00	35	34
	41.6	0.92	39	14
	56.0	0.98	45	33
From tapes made without wetting agent	23.0	0.98	55	36
	40.3	0.92	23	17
	50.2	0.85	15	14
	60.5	0.35	13	14
From extruded strands	33.4	0.99	35	41
	33.6	0.99	35	40
	34.1	0.98	44	50
	34.8	0.96	34	41

LIST OF FIGURES

- Figure 1. The modified Couette apparatus used for fiber alignment:  
a) motor, b) rotating disk, c) fixed rubber disk,  
d) screen, and e) vacuum.
- Figure 2. Granules made from 1/8" long, single short fibers:  
a) E-glass fibers, b) Thornel 25 graphite fibers, and  
c) boron fibers.
- Figure 3. Flexural strength and modulus of the glass fiber/epoxy composites molded from granules: O, 1/8" fibers;  $\Delta$ , 1/4" fibers;  $\square$ , 1/2" fibers. The solid lines are calculated from Eqs. 1a and 1b. <sup>2</sup>The solid curves (AM) are Anderson and Morris's data.
- Figure 4. Flexural strength and modulus of the glass fiber/epoxy composites molded from impregnated tapes: the symbols are the same with those in Figure 3. The dotted curve represents the strength level for the composites molded from granules.
- Figure 5. Flexural strength and modulus of the graphite fiber/epoxy composites molded from granules: O, hand laid-up granules;  $\bullet$ , extruded strands. The straight lines are calculated from Eqs. 1a and 1b.
- Figure 6. The coordinate system used for determination of fiber orientation.
- Figure 7. Cumulative fiber orientation distributions observed in typical glass fiber/epoxy composites: O and  $\bullet$ , for 61.3 and 40.4 vol. % composites, respectively, molded from granules;  $\square$  and  $\blacksquare$ , for 40.8 and 38.0 vol. % composites, respectively, molded from impregnated tapes; the broken curves, for a 59.2 vol. % composite molded from the extruded strand of encapsulated, bundled fibers.
- Figure 8. Type of failure observed for composites molded from granules and impregnated tapes: A)  $v_f = 0.533$ ,  $E = 4.13 \times 10^6$  psi,  $\sigma = 79.5 \times 10^3$  psi, made from granules of 1/8" fibers; B)  $v_f = 0.555$ ,  $E = 5.84 \times 10^6$  psi,  $\sigma = 118.5 \times 10^3$  psi, made from thin granules of 1/8" fibers; C) The same with B); D)  $v_f = 0.491$ ,  $E = 4.31 \times 10^6$  psi,  $\sigma = 84.2 \times 10^3$  psi, made from impregnated tapes of 1/2" fibers.

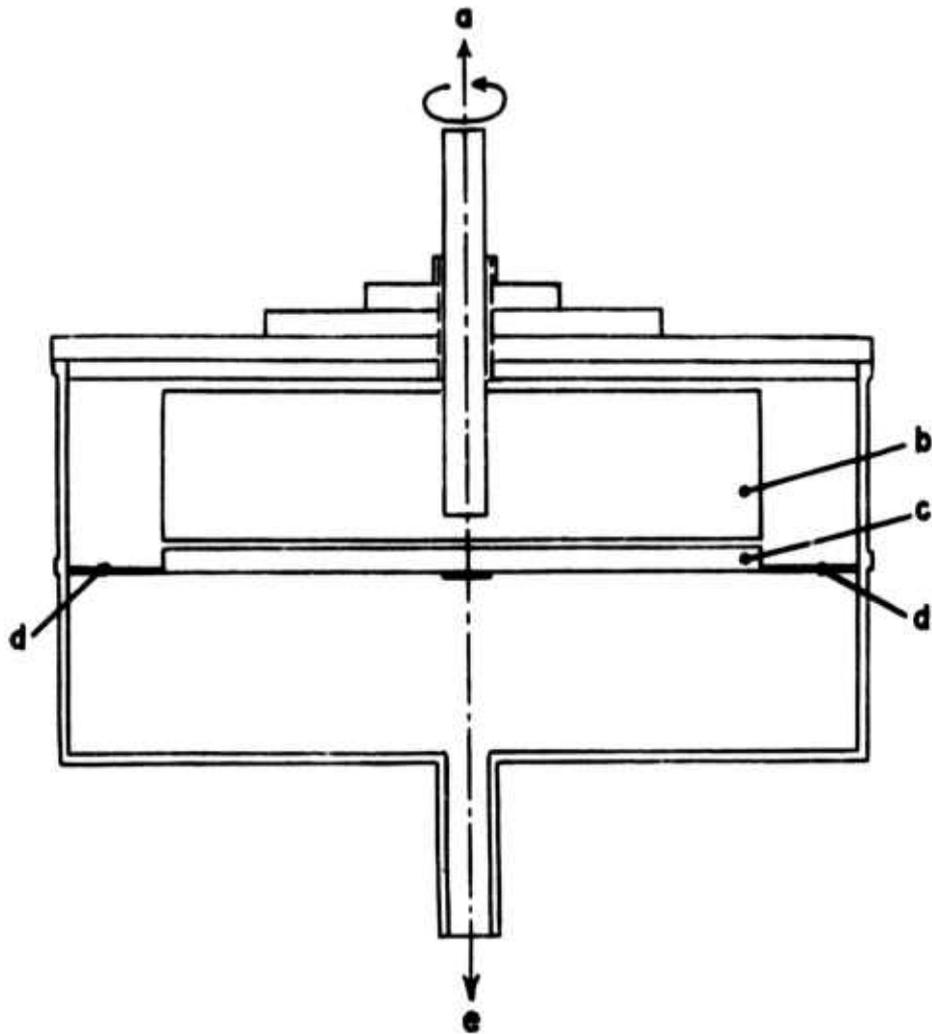
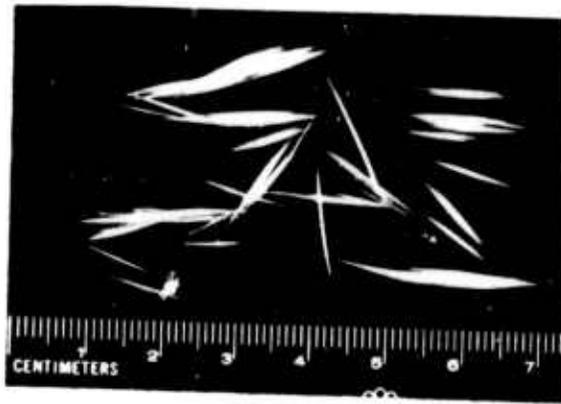
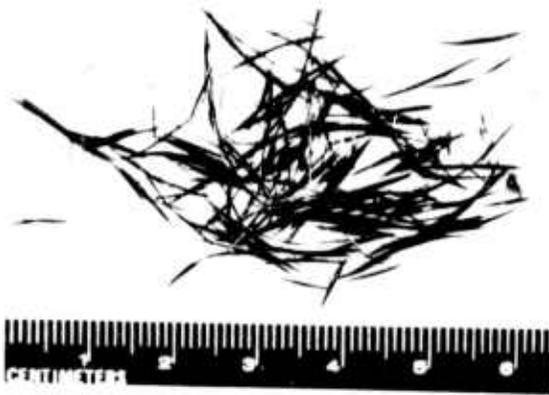


Figure 1

A



B



C



Figure 2

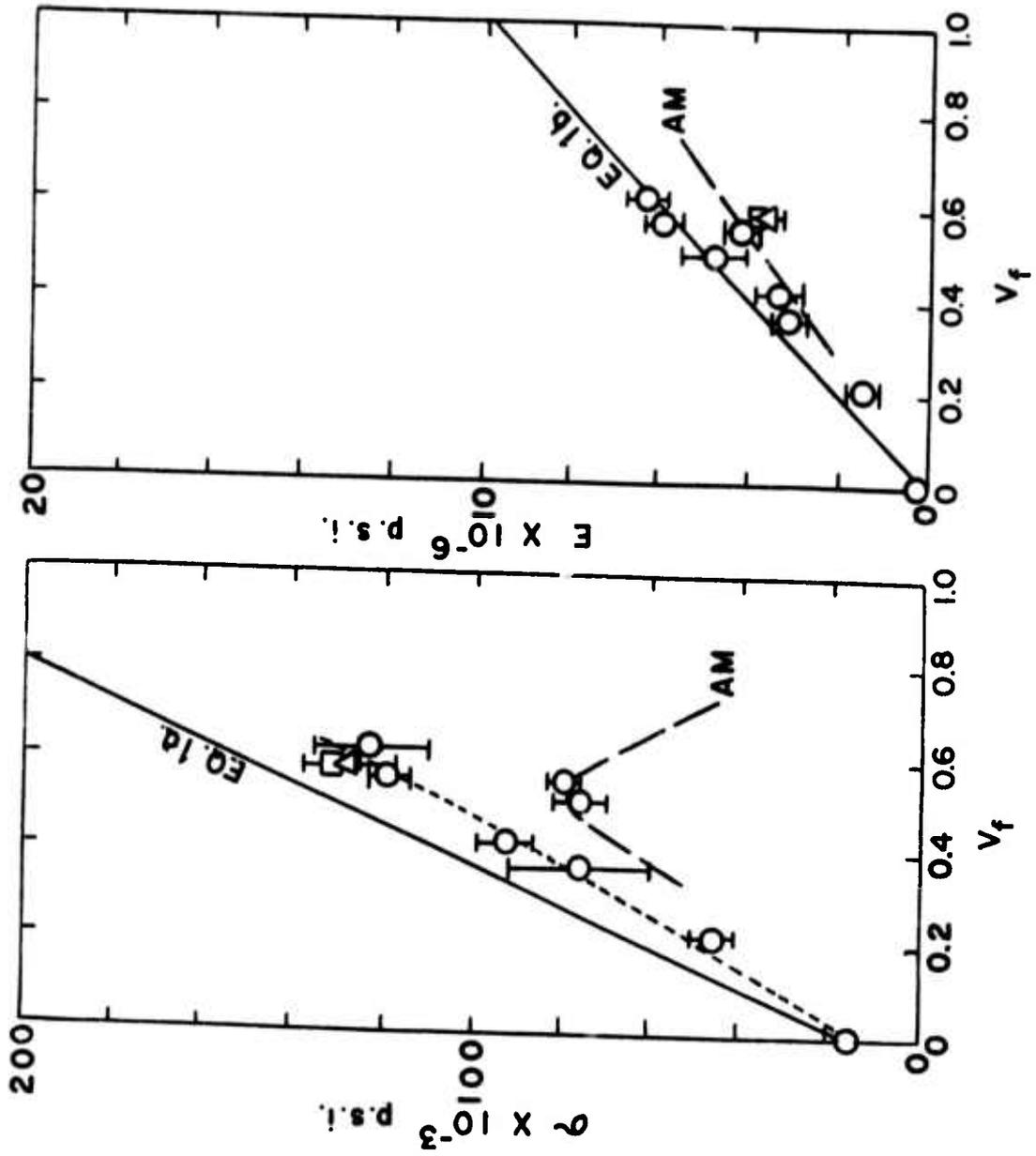


Figure 3

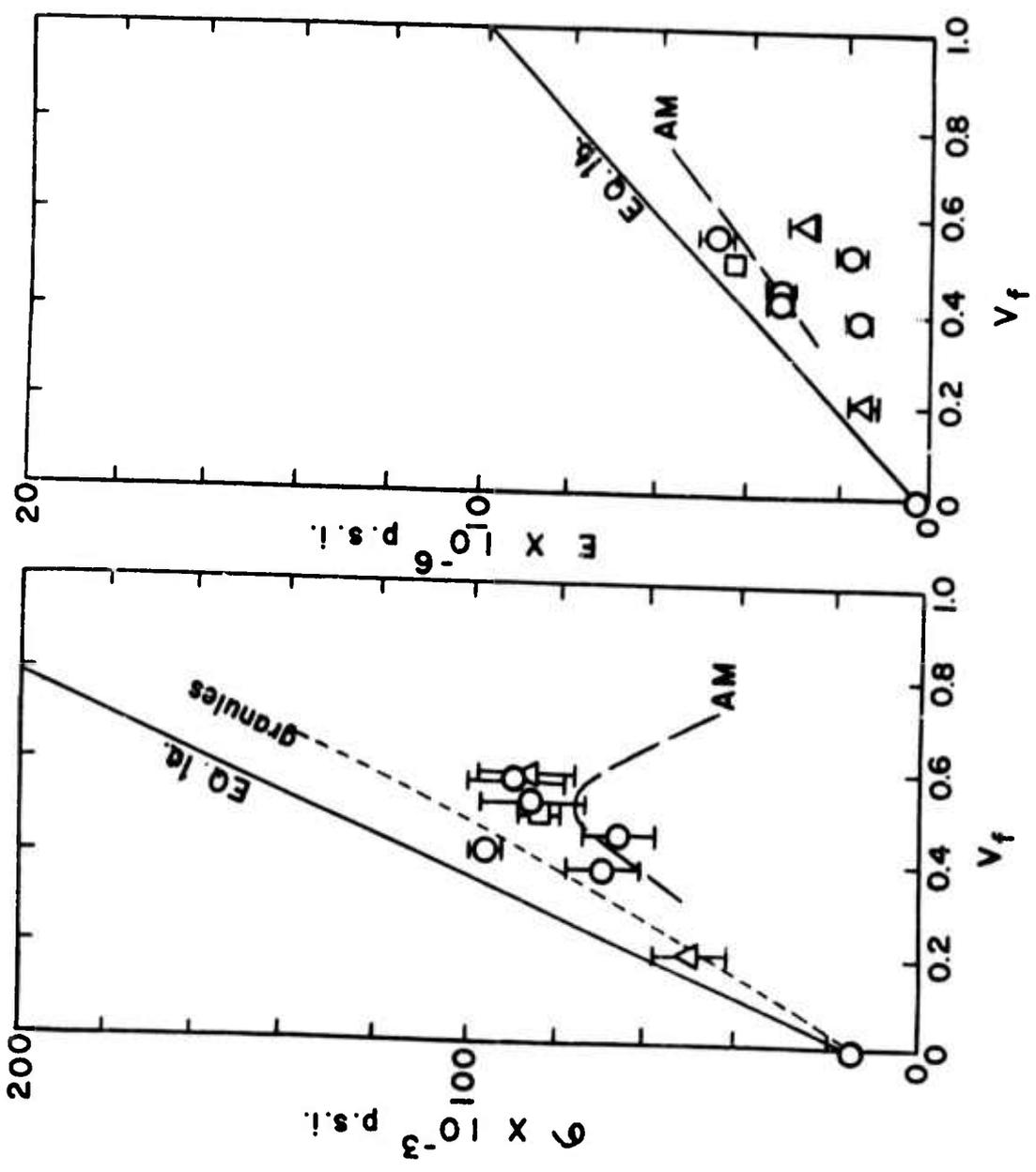


Figure 4

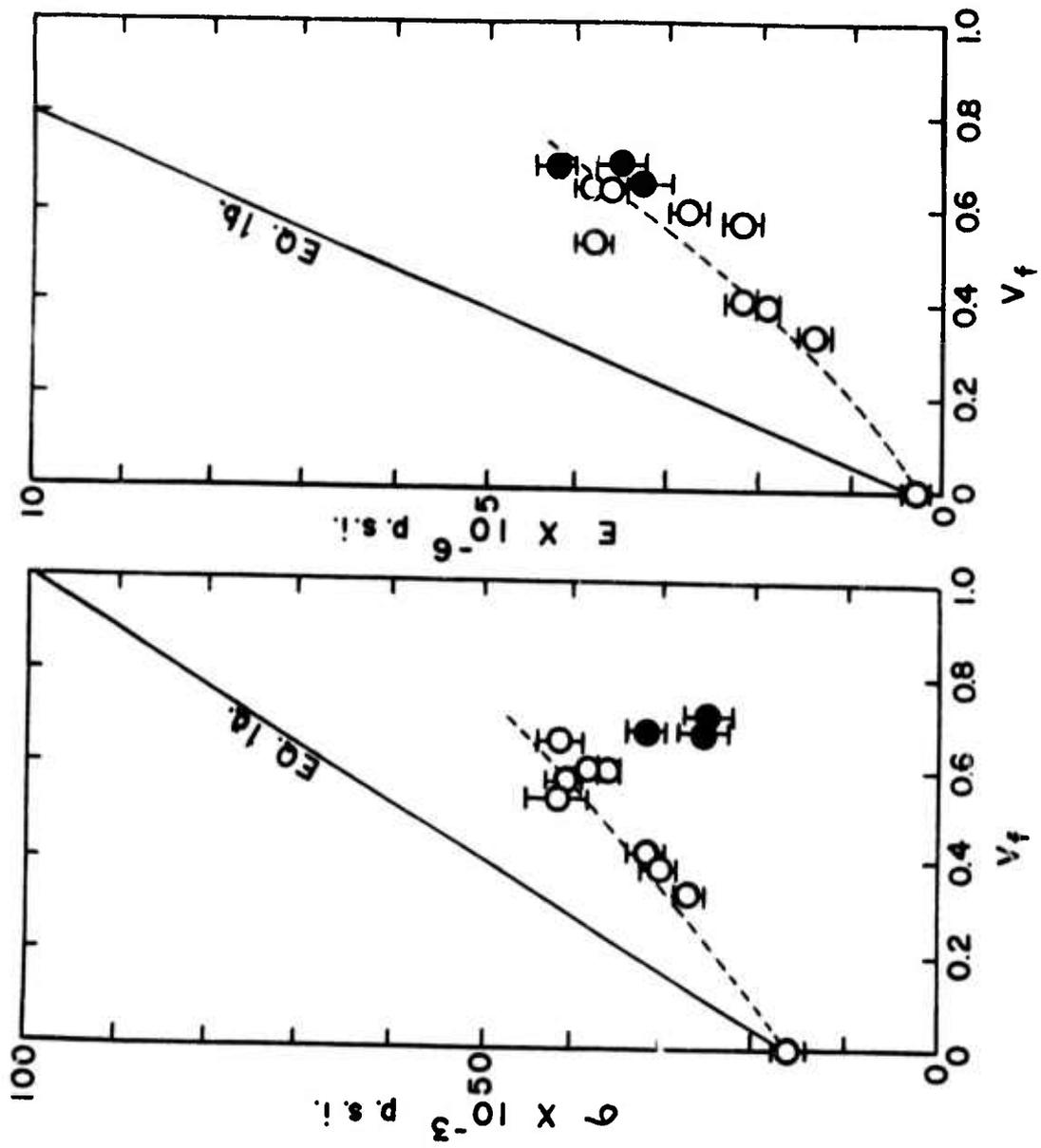


Figure 5

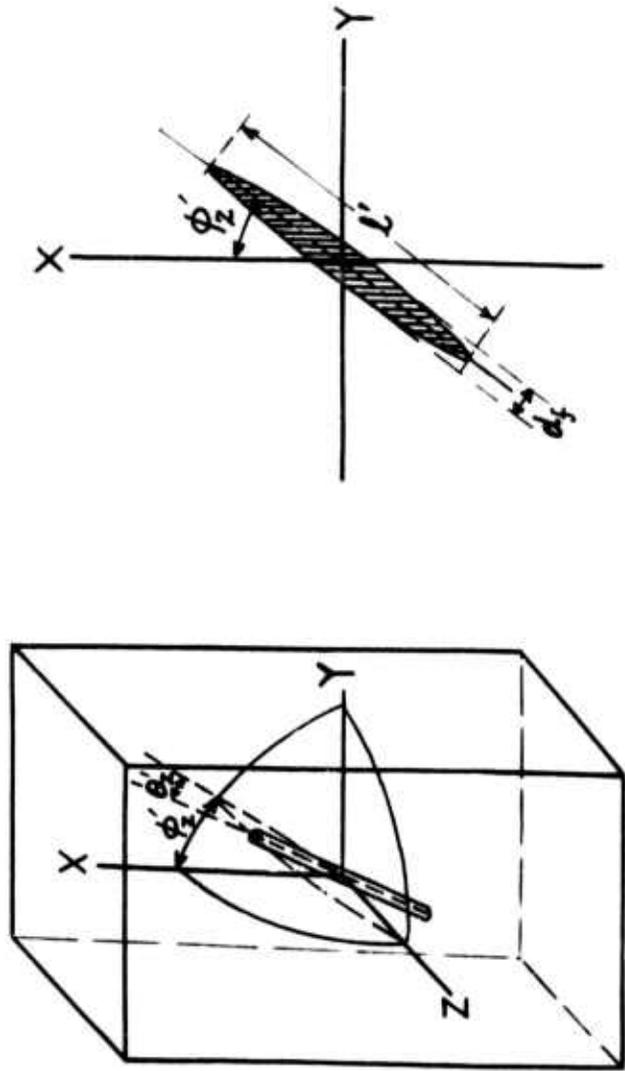


Figure 6

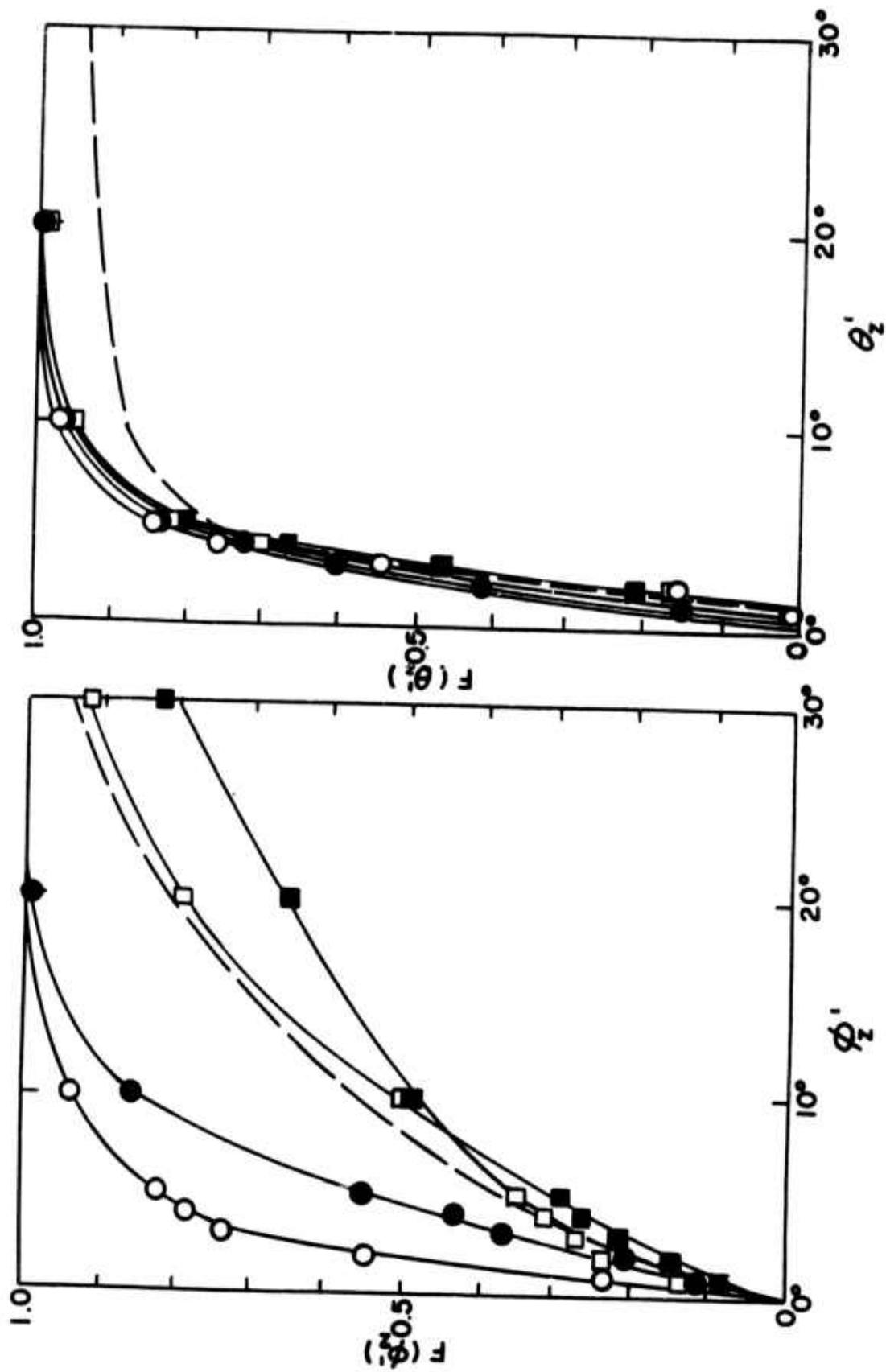


Figure 7

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D



C



B



A

Figure 8