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METHODS FOR AN INVESTIGATION OF THE EFFECT OF MATERIAL COMPONENTS ON THE MECHANICAL CHARACTERISTICS OF GLASS-FIBER-REINFORCED PLASTICS

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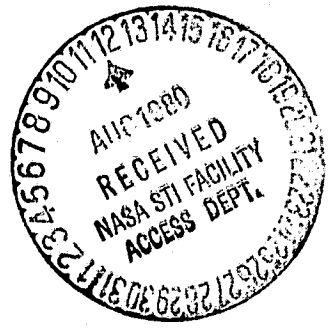

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16. Abstract
The materials used in the production of glass-reinforced plastics are considered, taking into account matrix polyester materials, the reinforcing glass materials, and aspects of specimen preparation. Various methods of investigation are discussed, giving attention to optical impregnation and wetting measurements and the gravimetric determination of the angle of contact. Deformation measurements and approaches utilizing a piezoelectric device are also considered.

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METHODS FOR AN INVESTIGATION OF THE EFFECT OF MATERIAL COMPONENTS ON THE MECHANICAL CHARACTERISTICS OF GLASS-FIBER-REINFORCED PLASTICS

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Good adhesion between the glass fibers and the plastic is especially/178* important for the quality of the compound. Prerequisite is a sufficient impregnation of glass fiber-reinforcement and the greatest possible wetting of the individual glass fibers by the plastic. The influence of impregnation and wetting on the mechanical characteristics of the compound have not previously been investigated.

1. Introduction

Glass-reinforced plastics are compounds of strong inorganic glass fibers and organic cast resins. The manufacture of the material and the shaping of the desired components are carried out simultaneously. The mechanical characteristics of this material are defined by

1. the weight and volume portions of the individual components,
2. the mechanical characteristics of the individual components,
3. the type of glass-reinforcement and
4. the adhesion between glass and resin.

Good adhesion is of special importance for the quality of the compound material. A prerequisite for this adhesion is a sufficient impregnation of glass-reinforcement and the greatest possible wetting of the individual glass fibers by the cast resin during production. Only a few investigations have been carried out on this subject. Information is completely lacking on the effect of impregnation and wetting on the mechanical characteristics of glass-reinforced plastics (GRP). There is also very little information on acquisition and evaluation of primary

*Numbers in the margin indicate pagination in the foreign text.

damage due to mechanical stresses, which may occur in the form of microcracks in the boundary surface area between glass and matrix.

Standard resins are mainly preferred in the technical production of GRP. Practically nothing is known about the effect of differing shaping behavior of the resins on the mechanical characteristics of the compound material. Therefore it is logical to conduct fundamental impregnation, wetting and shaping investigations of GRP with resin mixtures, composed of a standard resin with the addition of different amounts of a resin for increasing flexibility.

2. Materials Employed

2.1 Matrix Materials

The unsaturated polyester resins listed in Table 1 were employed as matrix materials. In addition mixtures of Palatal P5 and Palatal E210 were examined.

Table 1: Composition of the Past Resins Investigated

Type of Resin	Manufacturer	Abbreviated Designation	Viscosity CP
Palatal P5	BASF AG, Ludwigshafen	UP-P5	1000 to 1200
Palatal E210	BASF AG, Ludwigshafen	UP-E210	400 to 600

Palatal P5 is a resin of average reactivity and viscosity, hardening relatively rapidly and has a variety of applications. After the addition of suitable hardeners this material is totally converted by means of mixed polymerization under normal pressure into the rigid, cross-linked state and can therefore not be melted or dissolved.

Palatal E210 is a resin of medium to low viscosity and low reactivity, which is converted by means of mixed polymerization after the addition of suitable hardening agents to the transparent, flexible state. UP-P5 and UP-E210 may be mixed, UP-E210 contributing flexibility to the mixture.

The hardening proceeds more slowly and with less heat than in the case of pure UP-P5.

Palatal-Catalysator-Paste (manufacturer BASF AG, Ludwigshafen) was employed as hardening agent, a suspension of a cyclohexanonhydroperoxide mixture in phtalic acid ester. The peroxide content amounts to approx. 40% and the content of active oxygen approx. 5%. Table 2 provides information on the resin mixtures employed, the hardening agent, processing temperature, hardening temperature and hardening time.

Table 2: Composition of the Examined Mixtures of UP-P5 and UP-E210.

UP-P5 Gew.-%a	UP-E210 Gew.-%a	Härter Gew.-%a	b Verarbeitungstemperatur °C	c Härtungstemperatur °C	d Härtungszeit h
100	0	2	e RT	140	3
90	10	2	RT	140	3
80	20	2	RT	140	3
75	25	2	RT	140	3
70	30	2	RT	140	3
60	40	2	RT	140	3
50	50	2	RT	140	3
25	75	2	RT	140	3
0	100	2	RT	140	3

Key:
a. weight in %
b. processing temperature
c. hardening temperature
d. hardening time
e. room temperature

Table 3: Composition of the Glass-Reinforcements Employed /179

Tafel 3. Zusammenstellung der verwendeten Glasfaserverstärkungen.

a Glasfaserverstärkung	b Typ	c Hersteller	d Schlichte	e Binder	f Kurzbezeichnung
Spinnfaden g	K 92 -ES 10-400	Gevetex-Textilglas GmbH, Düsseldorf	i Silan	–	GSSF – K 92
Spinnfaden g	K 23 -ES 10-400	Gevetex-Textilglas GmbH, Düsseldorf	j Chrom	–	GSSF – K 23
Matte h	M 113-ES 10-100	Gevetex-Textilglas GmbH, Düsseldorf	k Methacrylsilan	l 3 bis 4% (in Styrol löslich)	M 113 – 100
Matte h	M 212-ES 10-400	Gevetex-Textilglas GmbH, Düsseldorf	i Silan	m 8 bis 12% (in Styrol schwer löslich)	M 212 – 400
Matte h	M 312-ES 10-400	Gevetex-Textilglas GmbH, Düsseldorf	i Silan	n 12 bis 16% (in Styrol schwer löslich)	M 312 – 400

Key:
a. glass reinforcement
b. type
c. manufacturer
d. facing
e. binder
f. short description
g. threads
h. mat
i. silane
j. chromium
k. methacrylsilane
l. 3 to 4% (soluble in styrol)
m. 8 to 12%(difficult to dissolve in styrol)
n. 12 to 16%(difficult to dissolve in styrol)

2.2 Strengthening Materials

The commercially available glass threads and mats listed in Table 3 were employed as strengthening materials.

2.3 Production of Probes

The pure resin probes were produced in cast forms of aluminum, consisting of three parts, two cover plates and a u-shaped core.

For the manufacture of probes strengthened in one direction a winding machine was employed, with which the impregnated glass threads were wound onto an octagonal with a total of seventeen layers. Once the octagonal winding was completed, it was covered with metal plates, which were screwed on. By this method a smooth surface of the hardened laminae was obtained after hardening. The glass threads and resin mixtures employed are listed in Table 4.

Table 4: List of the Probes Examined, Strengthened in One Direction.

UP-P5 Gew.-%a	UP-E210 Gew.-%a	Spinnfaden b	Glasgehalt Gew.-%c
100	0	GSSF – K 92	51,9
90	10	GSSF – K 92	51,1
80	20	GSSF – K 92	53,8
70	30	GSSF – K 92	54,9
60	40	GSSF – K 92	52,3
50	50	GSSF – K 92	54,8
100	0	GSSF – K 23	54,0
90	10	GSSF – K 23	50,1
80	20	GSSF – K 23	49,7
70	30	GSSF – K 23	49,8
60	40	GSSF – K 23	48,0
50	50	GSSF – K 23	53,7

Key:
a. weight in percent
b. threads
c. glass content

The mat reinforced probes were produced in a hot-wet-pressing process. All test plates (Table 5) were 300 mm x 300 mm x 4 mm in size. In all cases 2% hardening agent was added to the resin or the resin agent. The pressing temperature was 100°C, pressing time was ten minutes and pressure was 200 N/cm^2. All plates were subsequently hardened for 15 hours in the oven at 100°C.

Table 5: List of the Pressed Laminae Examined

UP P5 Gew. %	UP E210 Gew. % a	b Glasfaserverstärkung	c Glasgehalt a Gew. %
100	0	M 113-100	42,1
75	25	M 113-100	42,5
50	50	M 113-100	44,5
25	75	M 113-100	42,9
0	100	M 113-100	43,6
100	0	M 212-400	42,5
75	25	M 212-400	41,3
50	50	M 212-400	43,1
25	75	M 212-400	41,7
0	100	M 212-400	44,8
100	0	M 312-400	43,3
75	25	M 312-400	42,6
50	50	M 312-400	43,5
25	75	M 312-400	42,7
0	100	M 312-400	44,7

Key:
a. weight in percent
b. glass reinforcement
c. glass content

3. Methods of Investigation

3.1 Optical Impregnation and Wetting Measurements

The increase in light translucency of glass fiber products after immersion in liquid cast resin is utilized as a measure for impregnation and wetting. This is based on the fact that a parallel beam of light loses less in intensity through refraction and reflection, the further the wetting proceeds. If light passes from one medium into another at a defined angle, it is refracted according to the Snellius law of refraction when the two media exhibit different indices of refraction. If a parallel beam of light falls on a glass fiber product with circular cross-section of individual fibers - the indices of refraction of E-glass and air vary by about 0.5 - the light hits the glass surface at different angles, with the exception of the peak point of the fibers, so that the light is refracted and reflected. Therefore only a fraction of the primary beam passes through the glass fiber, i.e. it is relatively opaque. If on the other hand the glass fibers are surrounded by resin, with an index of refraction approximately equal to that of the E-glass, the light is refracted and reflected only to a small degree and the system resin-glass is more translucent than the glass fiber alone.

The experimental equipment is shown in Figure 1. Two experimental lamps (1) of the same type are arranged adjacent to one another in an opaque sheet metal box. Under each lamp at a distance of 25 cm

there is a selenium photo element (5) with a diameter of 40 mm. The two lamps are fed individually by a constant voltage source (6), because the measurements would be falsified by changes in light intensity due to the smallest deviations in circuit voltage. This equipment makes it possible to adjust the lamps to exactly the same light intensity. The lowering device permits external control of the immersion of the small frame with the glass fiber product parallel to the resin surface into this resin. A ring on the bottom of the resin container prevents the glass-reinforcement from coming in contact with the container bottom. /180

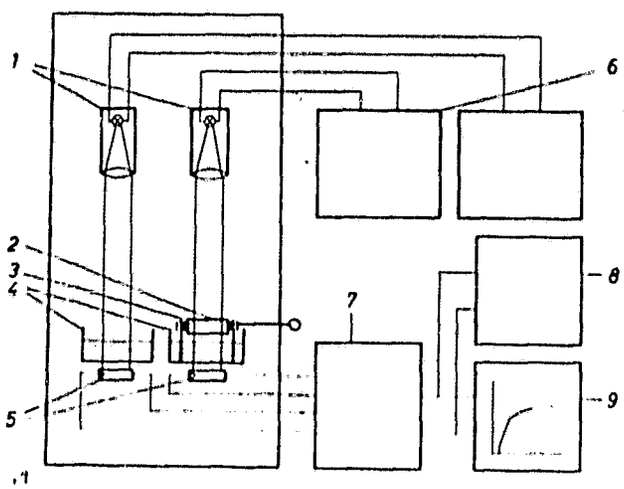

Figure 1: Schematic design of the optical impregnation and wetting device.

1. experimental lamps
2. small frame with probe
3. lowering device
4. resin container
5. selenium photo elements
6. constant voltage sources
7. dual current-voltage converter with differential amplifier
8. voltmeter
9. compensation recorder

For the mat probes pieces 80 mm x 75 mm in size were employed, which were clamped on small frames made of aluminum (square inner dimensions 50 mm x 50 mm, outer dimensions 60 mm x 55 mm, height 30 mm, weight 0.51 N). These frames were blackened by means of electrolytic oxidation to avoid irritating reflection. The thin glass threads were wound onto a square core, consisting of four aluminum frames screwed together. The threads were glued to the wound core and the screws were then removed. The upper portion of Figure 2 shows one-half of a wound core, in the middle are two frames with threads and at the bottom there are two frames, one with a mat and one with woven material. In the examinations of threads in each case frames with four layers of threads on top of one another were used.

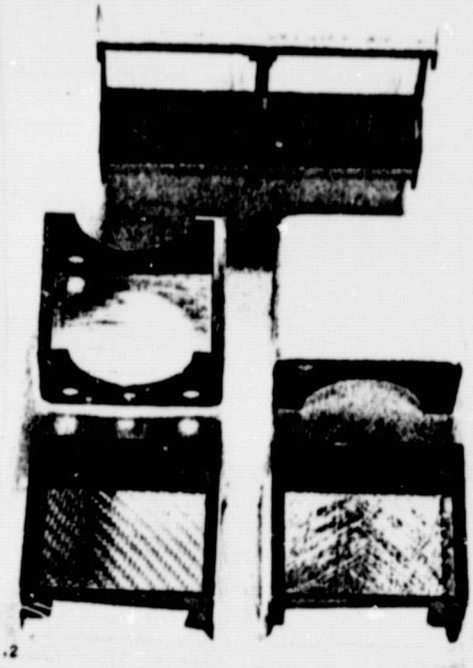

Figure 2: Probe core for the optical impregnation and wetting measurement.

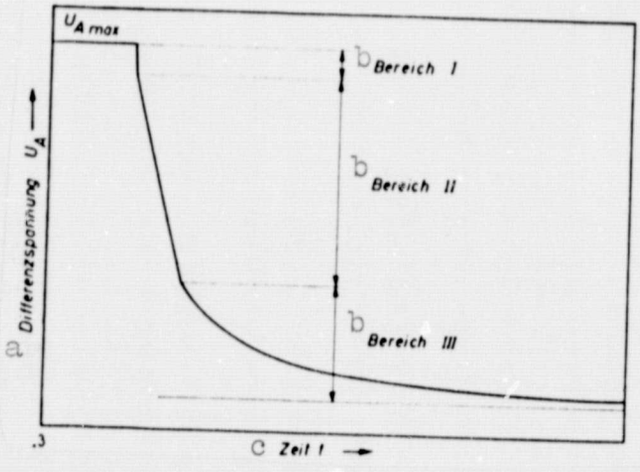

Figure 3: Schematic curve representation in the optical impregnation and wetting measurement.

Key:
a. voltage difference
b. area
c. time

At the beginning of a trial the measuring system was adjusted to zero (i.e. voltage difference $U_A = 0$), after the containers with the resin to be studied was introduced into the two beam paths. The frame with glass-reinforcement was subsequently positioned in one of the beam paths on the lowering device, reducing the light intensity falling on the selenium photocell underneath. The voltage difference $U_{A\,max}$ then

shown is a measure for the reduction of the light by the glass fiber product. When the frame with the glass-reinforcement was lowered into the resin, the curve shown in Figure 3 resulted. It may be divided into three areas:

Area I:
The curve follows a linear course and characterizes the sinking of the frame into the resin. This process is characterized by a simultaneous impregnation and wetting.

Area II:
The curve also follows a linear course with a slight upward inclination. The resin permeates the glass-reinforcement from beneath. This process is concluded when the resin has completed climbing within the frame. Impregnation with simultaneous wetting has been achieved.

Area III:
The curve follows a bent course and approaches a final value. A pure wetting process has been achieved.

In order to compare the curves made for different types of resin and glass-reinforcements, the impregnation and wetting degree was defined as

$$B = \frac{U_{A\max} - U_A}{U_{A\max}} 100\%$$

B_{10} is the degree of impregnation and wetting, obtained during a trial of ten minutes.

U_A becomes equal to $U_{A\ max}$ when the glass has not yet been touched by the resin. In the case of $U_A = U_{A\ max}$, B = 0%, in the case of U_A = 0, B = 100%. If B, the degree of impregnation and wetting, is plotted against time t, the curve follows the same course in principle as in Figure 3 with the three areas. /181

A minimum of five trials was carried out for each glass-resin combination. The trial duration was limited to ten minutes, corresponding

approximately to usual processing times.

3.2 Gravimetric Measurement of Boundary Angles and Wetting Procedures

The measurement of boundary angles between cast resin and glass as well as the measurement of wetting procedures between mats or woven materials and cast resins may be carried out with a gravimetric measurement method. In this case the weight change, occurring when the test core is drawn out of the resin or when the core touches the resin, is determined as a function of time. The test core is suspended on a scale beam over a container with resin. It must be possible to move the vibration-proof resin container up and down continuously.

The trial equipment shown in Figure 4 consists of a microscale (type 4162, Sartorius Company) with a weight range of 200 mg and a table (4) for the measuring liquid container (3), which can be moved up and down slowly and continuously by an electromotor (8) via a spindle (5). A clamp (2), hung on one side of the scale beam (1), serves to hold the test core. Weight changes occurring when the test core touches the measuring liquid or when it is drawn out of this liquid are indicated on the electric control device (9) of the scale and can be recorded by the compensation recorder (10) as a function of time. The actual measuring system is housed in a plexiglass covering in order to exclude inaccuracies in the measurement due to air motion, dust, etc.

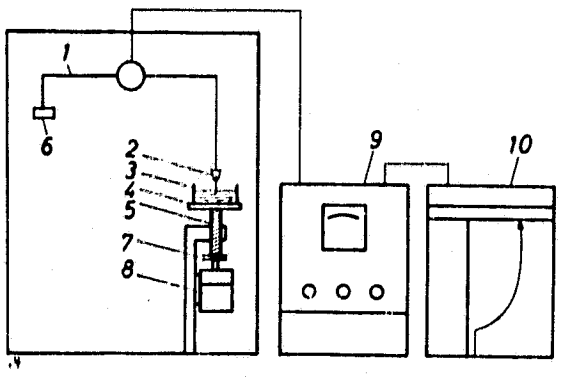

Figure 4: Schematic representation of the gravimetric wetting device.

Key:
1. scale beam
2. clamp
3. measuring liquid container
4. table
5. spindle
6. counterbalance
7. clutch
8. electromotor
9. electronic control device
10. compensation recorder

For measurement of the boundary angle ϑ , in the contact of elementary glass fiber with the liquid cast resin, approx. 30 to 40 mm long fibers were drawn out of the threads of the reinforcing glass under study with the aid of the microscope. These were subsequently hung on the microscale and brought into contact with the measuring liquid by means of vertically shifting the table carrying the measuring liquid. The change in weight ΔG is shown in Figure 5 as a function of time. The final value ΔG_E was used for calculation of $\cos\vartheta$. The following equation applies:

$$\cos\vartheta = \frac{\Delta G_E \cdot 981}{U\,\sigma_F}.$$

In this case ΔG_E is the final value of weight change in p,U is the circumference of fibers in cm and σ_F is the surface tension of the measuring liquid in $\frac{\text{dyn}}{\text{cm}}$.

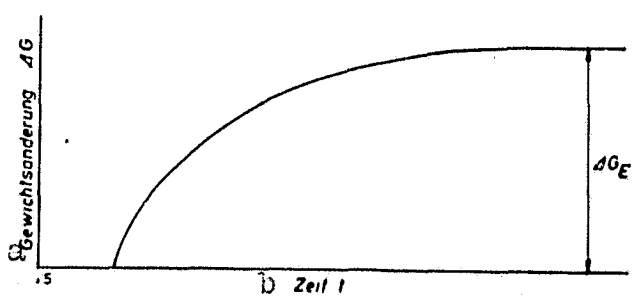

Figure 5: Schematic representation of course of the curve in boundary angle measurement.

Key:
a. weight change
b. time

Corrections for lift were not required because of the small volume of the immersed glass fibers. Due to the small diameter D of the elementary fibers (D = 8 to 15 µm) a microscopic measurement is not sufficiently exact for the necessary measurement of fiber circumference. The fiber diameter was therefore also determined by gravimetric means. For this purpose the fibers were brought into contact with a completely wetting test liquid (i.e. $\vartheta = 0°$ or $\cos\vartheta = 1$) with a known surface tension and ΔG_E was determined.

The same procedure as described above may be applied to the determination of the wetting behavior of the glass fibers, mats and woven pieces. In the conduction of trials ΔG-t curves were made for the immersion of the glass probes in liquid resin. For these studies elementary fibers 30 mm long and mats 30 mm long and 6 mm wide were used. For comparison of various glass-resin systems it was necessary to relate the weight increase registered as a function of time to a quantity characterizing the reinforcing glass. In the case of the elementary fibers and threads the circumference U of the fibers lends itself as a basis for comparison, while in the case of mats the macroscopic cross-sectional area of the probe may be considered without taking into account the structure of reinforcement. Exact measurements of mat thickness, however, proves to be too difficult. Since probes of the same length and width were always used in the studies and only the thickness differed from one reinforcement type to the next, the weight increase during wetting trials is related to the beginning weight of the probes. The calculated thickness D^x in mm is proportional to the probe weight /182 according to the equation

$$D^x = \frac{1}{L B \gamma} G$$

Here: L is the probe length in mm, B is the probe width in mm and γ is the specific weight of the glass.

3.3 Shaping Measurements

An electronic universal testing machine was employed for tensile trials. Contact jaws adjustable in pressing force were used to hold the probes in these trials. The upper contact jaw was connected to the electric system for measurement of force, supplying an electric voltage signal proportional to the force, which was registered against time by a recorder. The lower contact jaw was connected to the transverse yoke, which was moved up and down at constant speed by a mechanical spindle drive.

The tensile trials were carried out and evaluated according to DIN 53455. In addition the proportionality limit was determined from the

tensile stress-strain diagrams. The proportionality limit is understood as the stress, up to which the stress-strain curve coincides with its tangent passing through the coordinate origin.

3.4 Direct-Contact Vibration Measurements

When microcracks are formed in GRP laminae subjected to strains, the stored elastic deformation energy is consumed in the formation of fracture surfaces, in plastic shaping in the fracture zones and in excitation of elastic vibrations. A piezoelectric vibration transformer placed on the test core can convert these vibrations into electric voltage, which can be registered with a suitable measuring device as a function of time.

The measuring device in Figure 6 consists of a piezoelectric vibration transformer (2), an impedance transformer (3), a sound impulse level meter (4) and a compensation recorder (5).

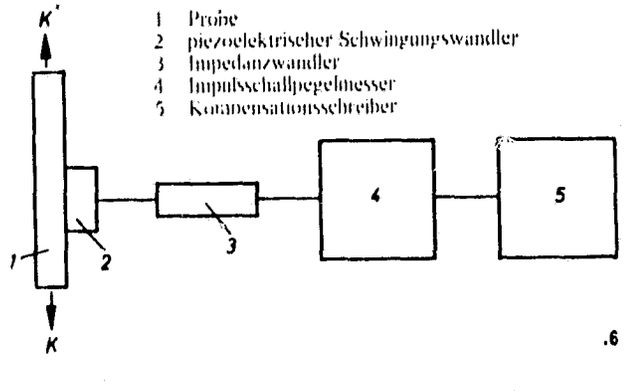

Figure 6: Schematic representation of the direct-contact vibration measurement device.

1. probe
2. piezoelectric vibration transformer
3. impedance transformer
4. sound impulse level meter
5. compensation recorder

In the trials the piezoelectric vibration pick-up was fixed to the probe by means of adhesive wax and the direct contact vibration was registered as a function of time. The first reading from the vibration intensity-time curve is evaluated under the assumption that this is an indication of microcrack formations, i.e., damage begin. This first reading was converted into the corresponding stress value $\sigma_{\ddot{u}}$, or the strain value $\varepsilon_{\ddot{u}}$. The trial results will be published separately.