PROGRESS REPORT VIII

(December 1984 - February 1985)

De-icing and Prevention of Ice Formation of/on Offshore Oil-Drilling Platforms

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De-Icing and Prevention of Ice Formation of/on Offshore Oil Drilling Platforms (DACA 84-83-00031)

(1) Polyethylene Foam Mat

A polyethylene foam mat (NALGENE "Clear Sheets," Nalgene 6281 series, closed cell, crosslinked (6mm thick) was cut into a 3" square piece. This was stuck to an Al-plate (3" square) with double stick scotch tape. This sample was then suitable for shear adhesion tests except that the sample was too thick for such tests, but an approximate, preliminary value could be obtained. The shear adhesive strength amounted to,

0.53, 0.48 and 0.34 kg/cm^2 Av. 0.45 kg/cm^2

The mat was sliced to about half its original thickness and samples were measured (thickness 3±0.3 mm).

Sample Treatment	Shear Adhesive Strength, kg/cm^2
Without water (gale) erosion	0.45
3 h Erosion	0.34
Additional 6h Erosion (total 19h)	0.28

Erosion did not affect the shear strength although the surface of the foam was wet with water. The latter fact always increased the adhesive strength of other materials.

Polyethylene foam was coated with LR-5630 (1g)/SF-1154(0.5g) in 12ml toluene solution and in silicone masonary sealer (silicone resin 5% in petroleum thinner 95%, sold by True Value Store). The coat was applied with a 0.2mm gap roller (coater). The LR-5630/SF-1154 coat could be quite easily peeled off the foam i.e. the adhesive strength between the coat and the polyethylene foam is quite low.

Silicone Masonary Sealer-Coated PE-Foam

	kg/cm^2
Without Erosion	0.25
After 3h Erosion	0.44

After 3h of erosion the surface of the sample was water repellent.

Sliced PE-Foam (3" square, 3mm thick) was adhered to an Al-plate with cyanoacrylate adhesive under compression:

	kg/cm^2
Without Erosion	0.61
Coated with SF-1154 and Eroded for 16h	0.21
After additional 16h of erosion (total 48h and dried for 1 day	0.67

All the above adhesive strength values are within the requested strength limit (i.e. 25psi or 1.76 kg/cm^2), which is very encouraging.

Polyethylene foam tape (3mm thick tape is available commercially)

coated with silicone masonary sealer or SF-1154 having pressure sensitive adhesive at its back appears to be a good system for de-icing.

(2) Dow Corning Varnish #997 (recommended curing time and temperature: 3h, 200°C.)

(a) Preliminary Experiments

An Al-plate was coated with #997 by flow-coating and heated on a hot plate. The ice/coat adhesive strength was measured (as always -10°C).

Sample No.	Sample Conditions	kg/cm^2
1	180°C 3h	3.85
2	Plus 240°C, 3h	4.24
2	" "	2.25*
3	223°C, 1h	2.44
3	" "	1.03*
4	220°C, 2h	1.40
5	225°C, 2h	4.24
5	" "	1.03*

*Also coated with silicone 0.2 (MW 340)

(b) Curing Conditions

As the shear adhesive data appreciably fluctuate, the curing conditions for #997 were examined in more detail.

997 was coated on Al with a coater (gap 0.4mm) and heated to 200 ±

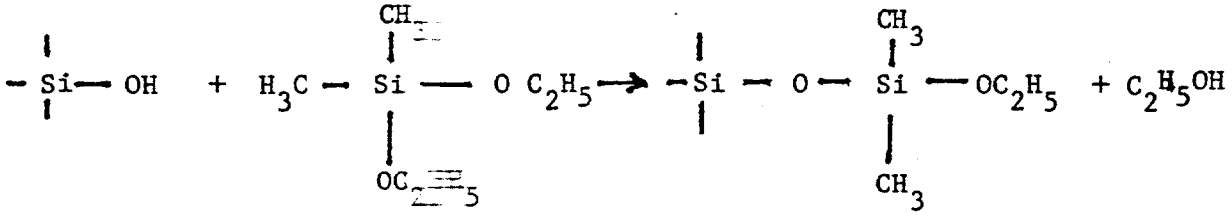
1.0°C for varying periods of time. The adhesive strength was then measured:

Heating time, h	1.5	3.0	4.0	4.5	5.0	6.0	7.5
Shear adhesive strength kg/cm^2	>4.74	>4.74	1.80	1.45	2.14	2.09	2.26

The adhesive strength as a function of curing time shows peculiar features, it decreases very rapidly at 4h, reaches a minimum at ca. 4.5h and then increases somewhat beyond this time.

A sample cured 4.5h (i.e. which shows the minimum adhesive strength as a function of curing time) was subjected to erosion for 16h. The adhesive strength amounted to 1.43 kg/cm^2 which lies under the requested value of 1.76kg/cm^2.

(c) #997 varnish was reacted with dimethyl-diethoxy silane. The assumed reaction is as follows:



The silane Me_2 Si $(Oet)_2$ functions as a chain extender was heated for 2h and was then coated with a coater (gap 0.4mm) on an Al-plate. The coat was heated at 200°C for 4.5h. After shear-testing it was further heated (i.e. total heating time 4.5h).

Sample #997/$Me_2Si(Oet)_2$ By Wt.	kg/cm^2 Cured at 200°C, 1.5h	Cured at 200°C, 4.5h
10/8	1.69	0.91
10/6	2.49	0.85
10/5	1.45	1.06
10/4	2.59	1.11
10/0	> 4.74	1.45

All values for the 4.5h-cured sample lie below the specified value (1.76kg/cm^2).

(2) The above adhesive mixture was further heated for 5h and coated on Al. The mixture contained some gel-particles and in the coat and many cracks after heating it at 200°C for 1.5h. The results are given below and are represented in Fig. 2.

Reacting #997 with $Me_2Si(Oet)_2$ did not improve the abhesion properties of the coat appreciably.

(c) Addition of Silicone Oil

Silicone oils poly(dimethyl siloxane)(MW 1x10^5) and G.E. SF-1154 were mixed with #997 varnish. The mixture was coated on an Al-plate and tested:

Sample	Composition #997/Silicone Oil By Wt.	Remarks (Appearance)	kg/cm^2
Polydimethyl Siloxane	10/0.5	silicone oil particles separated	0.86
"	10/1	" " "	0.68
SF-1154	10/1	uniform coat	0.31
"	10/2.5	film is very weak	----
"	10/5	" "	----

The #997 mix with silicone oil SF-1154 was subjected to erosion for 15h and stored at room temperature.

Time stored, days	0	10	11	12	14
kg/cm^2	1.06	1.94	1.76	0.77	1.31

the addition of SF-1154 (10 wt% to #997) only reduces the initial adhesive strength.

Generally the adhesive strength of #997 cured at 200°C for 4.5h was slightly below the specified value and did not change much with erosion.

(3) SR 80M Silicone Electrical Resin Made by G.E.

SR 80M consists of 34% silicone resin solution and changes to a tack-free film by drying at room temperature for 30 to 60 minutes.

The coating properties of SR-80M were poor compared with #997. It was difficult to obtain a smooth coat.

(a) Curing Experiments

A SR 80M film was coated on a glass-slide using an eye dropper. The coat was cured under a variety of conditions listed below and was immersed in toluene for a curing test for 2h.

Curing conditions	Toluene Curing Test
Room temperature , 30 min.	Dissolves
" " , 24 h .	Only a trace does not disolve
100°C , 1 h	Small amounts of film left
100°C , 2 h	Somewhat larger amount left but still small
100°C , 16 h	Large amount of film left

Results indicate that proper curing will take a long time probably 100 days or more, even as the varnish is a so-called room temperature curing varnish.

(b) Coating Experiments

SR 80M varnish was coated on two Al-plates which were heated to 100°C for 1h and 16h, respectively, the coats cracked when cooling to -10°C. Their adhesive strength was 1.31 kg/cm^2

(c) Modification of SR 80M

SR 80M (1g) and $Me_2Si(Oet)_2$ (0.1 g) were mixed and heated to ca. 60°C for 3h. However, no viscosity increase was observed.

(d) Addition of Silicone Oil SF-1154

SF-1154 (15% by weight) was added to SR 80M as a plasticizer. A clear solution was obtained. It was dried at room temperature for 2.5h and then heated at 100°C for 16h. A turbid coat was obtained. The coat cracked on

cooling to -10°C. Overall, SR 80M did not prove suitable for coating at low temperatures.

(4) Silicone PS 255 (Fluka Chemical Corp.).

PS 255 is a copolymer of dimethylsiloxane and 1% to 3% methylvinylsilane. It is cured by the addition polymerization with vinyl groups.

1.2g of PS 255 were dissolved in 7.2ml of toluene for obtaining a coating solution. This solution has a suitable viscosity for coating.

A coat was prepared on a glass-slide and heated at 100°C for two hours; however the coat dissolved completely in toluene during 1h at room temperature.

Next, a coat was heated at 150°C for 3.5h. A sticky, clear coat resulted. This coat only dissolved completely in toluene after 19h immersion. Thus PS 255 did not crosslink under these conditions.

A solution of 1g of PS 255 plus 0.004g of divinyl peroxide in 7ml of toluene was coated on a glass-slide and heated at 150° for 18h. The sticky coat was heated again, this time to 200°C for 1h. But crosslinking did not take place. It would take too long at this stage of the work to search for the proper crosslinking conditions.

(5) Vinyl-acrylate Copolymer Lacquer

It consists of 18% by wt. of polymer in a 82% solution in a petroleum

distillate. It is produced by the limited Gilsonite Laboratories under the trade name "Drylok". The coat is supposed to be curable at room temperature, experimental data are shown in Fig. 2. Shear adhesive strength increases with progressive curing; the coat increased in hardness.

A coat on an Al-plate (adhesive strength 0.98kg/cm^2 was exposed to erosion for 3h, its adhesive strength increased to 4.5kg/cm^2.

Adhesion between the co-polymer LR5630 and vinyl-acrylate co-polymer was found not to be suitable. Vinyl-acrylate is not satisfactory for our purpose.

(6) Dow Corning 3145 RTV Clear Adhesive-Sealant

It was cured at room temperature within 2h to a tack-free silicone rubber, while it was cured to a rubbery solid within 1/2 h (1/8" thick coat).

(a) Results for RTV silicone paste are given below.

Thickness of Coat mm	Shear Adhesive Strength kg/cm^2
0.4	0.26(1*) $\xrightarrow[3h]{\text{Erosion}}$ 1.79 (2 $\rightarrow$ 2.01 (12 $\rightarrow$ 7.08 (13) $\rightarrow$ 1.35 (14) $\rightarrow$ 1.16 (15) $\rightarrow$ 1.19 (16)
0.6	0.32 (2) $\rightarrow$ 1.14 (12) $\rightarrow$ 1.07 (13) $\rightarrow$ 0.77 (14 $\xrightarrow[3h]{\text{Erosion}}$ 1.33 (15) $\rightarrow$ 1.36 (16)
0.8	0.26 (2) $\rightarrow$ 1.06 (12) $\rightarrow$ 0.60 (13) $\rightarrow$ 1.02 (14) $\xrightarrow[3h]{\text{Erosion}}$ 1.15 (16)

The adhesive strength increases with elapsed time. i.e. with days of

curing. Erosion has an appreciable effect on coats cured for short times, but very little effect on long-time cured coats.

(b) RTV silicone plus Thomas silicone oil.

RTV-silicone paste was dissolved in toluene (27.5 by volume of paste), silicone oil was added and the mixture coated on Al-plates. (thickness of dried coat 0.11mm).

Results are given below:

RTV/Silicone Oil by Wt.	Adhesive Strength kg/cm^2
10/0*	1.00 (10) → 0.60 (11) → 0.56 (12) → 0.60 (14)
10/0	1.03 (1) → 0.61 (13)
10/1	0.86 (10) → 0.57 (11) → 0.79 (12) → 0.64 (14)
10/2	0.67 (10) $\xrightarrow[3h]{\text{Erosion}}$ 0.99 (11) → 0.88 (12) → 0.66 (14) → 0.76 (24)
10/4	0.57 (17) → 0.24 (3)
10/6	0.47 (1) → 0.28 (3)
10/8	0.28 (1) → 0.11 (3)
10/10	0.30 (1) → 0.13 (3)

*Coat-Thickness 0.2mm

The adhesive strength progressively decreases with increase in curing. This is contrary to the previous results. The addition of silicone reduced the adhesive strength. RTV silicone is being further investigated.

Conclusion

During this period's work the remarkably efficient deicing system polyethylene foam (Nalgene) was investigated. Other systems were also studied but were not suitable for our purpose except possibly for RTV silicone plus silicone oil, which is being investigated further.

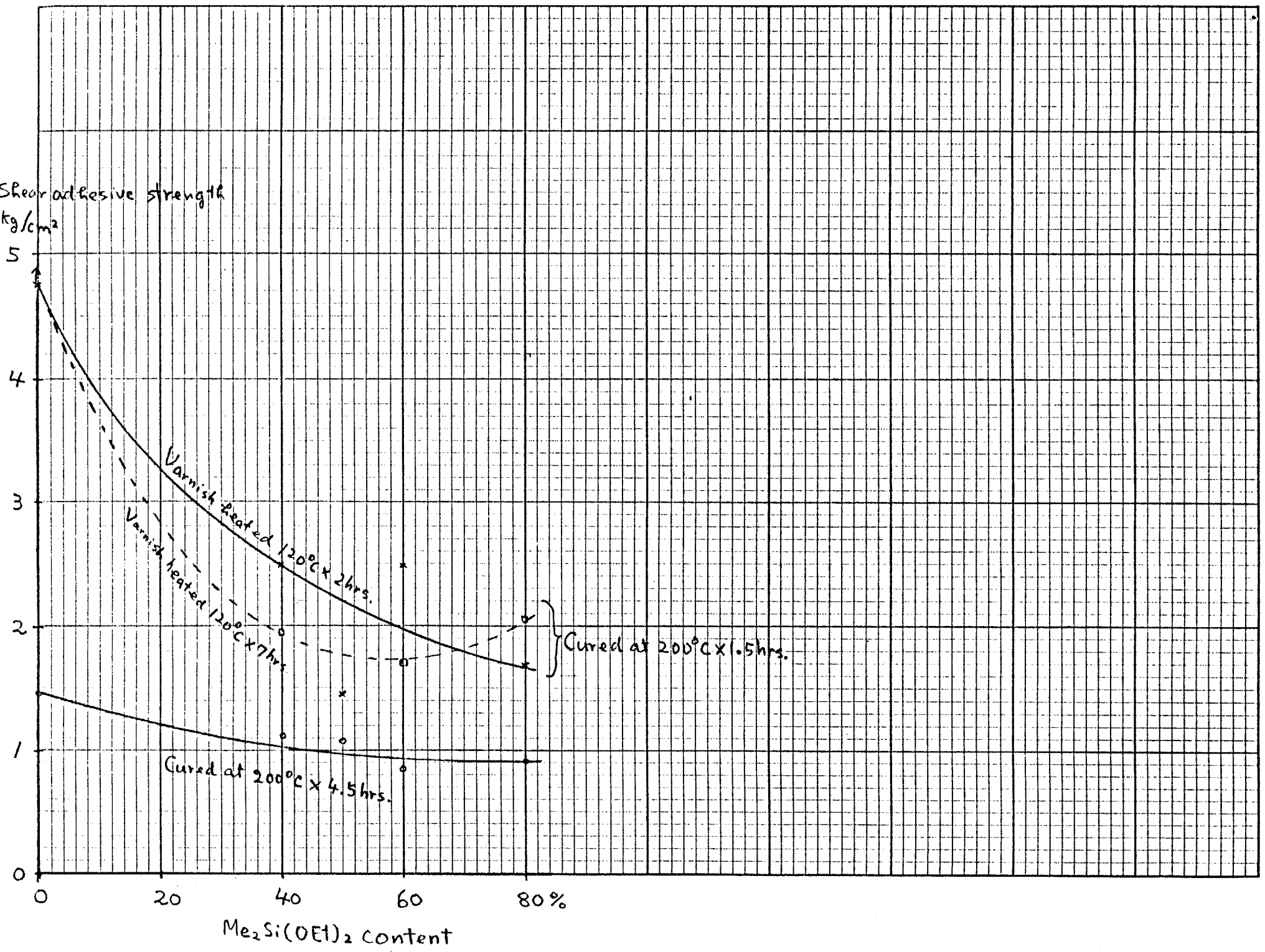


Fig. 1

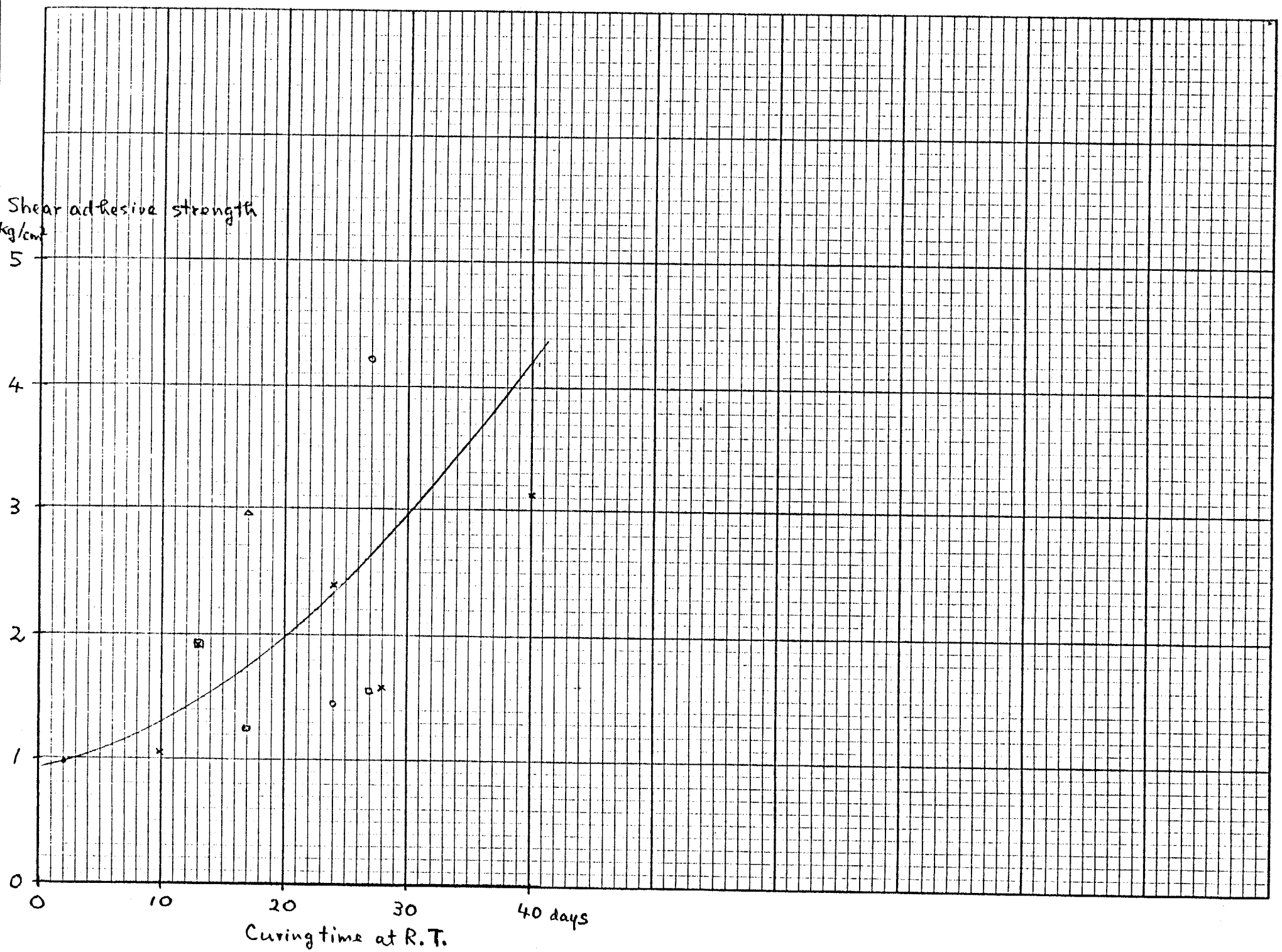


Fig. 2