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Irradiation Damage Studies of some Epoxy Resin Systems of Reduced Viscosity

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Science Research Council
Chemical Technology Group
Rutherford High Energy Laboratory
Chilton Didcot Berkshire
1972

SBN 90237647 0

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IRRADIATION DAMAGE STUDIES OF SOME EPOXY RESIN SYSTEMS OF REDUCED VISCOSITY

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ABSTRACT

Post irradiation mechanical properties are given for a number of epoxy materials, including some glycidyl amine resin systems having reduced viscosities.

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1 INTRODUCTION

The primary requirement for radiation stable organic materials lies in the construction of magnets for high energy physics applications. Many studies have already been made of the radiation stability of epoxy resin composites showing that some of these materials are able to withstand doses of 10^{10} rad without significant loss in mechanical properties.[1 - 4]

One important consideration in selecting an epoxy resin is that it should have a relatively low viscosity and long pot life and thus be suitable for impregnating closely wound magnet coils.

This report gives data on the radiation stability of further epoxy resin systems, including low viscosity MY 720* systems and the use of MY 720 as accelerator for anhydride cured epoxy resins. These materials are compared with the conventional tertiary amine accelerated MY 740*/HY 906* system. The effect of cure cycle on the radiation stability of two epoxy resin systems is also examined. In all cases the materials were prepared in the form of cast resins and glass reinforced composites.

2 SPECIMEN PREPARATION

2.1 The epoxy resins were cast into sheets of $\frac{1}{8}$" thickness and flexural test specimens of dimensions given in table 6 were cut from the sheets using a diamond tipped cutting disc.

2.2 The materials were also prepared in the form of glass fabric laminates of $\frac{1}{8}$" thickness using conventional wet lay-up techniques. Sufficient glass fabric to complete the experiment was cut into 9" squares, parallel and at 45° to the warp from 0.006" square weave glass fabric finished with an amino silane. Each laminate was constructed from 20 layers of the glass fabric such that the warp direction of each subsequent layer was rotated clockwise through 45°. This technique ensures that the finished laminate will show little directionality of properties due to differences between the warp and weft direction of the fabric. After complete wetting-out of the

*Ciba-Geigy (UK) Limited, Plastics Division, Duxford, Cambridge. Supplied resins and hardeners used in these experiments.

final fabric layer, the laminate was pressed between stops, to give a nominal glass/resin ratio of 60/40, and cured in the press at the appropriate temperature. The actual glass/resin ratio was determined for each laminate and these results are included in table 11.

2.3 The formulations of the epoxy resin systems tested in this report are given in tables 1 - 4 and details of cure cycles are given in table 5.

3 TEST PROCEDURE

The mechanical properties of both cast and laminate specimens in flexure were determined using three point loading applied by means of a commercial testing machine*. The test details are given in table 6. This test procedure is in accordance with that recommended for both sheet material and high strength reinforced plastic composites in ASTM D 790/66.

4 IRRADIATION

4.1 All specimens were irradiated using the spent reactor fuel element irradiation unit at UKAEA Harwell, Berks. This facility provides gamma radiation with a mean energy of 1.0 MeV at dose rates up to a maximum of 6 megarads per hour. Calibration is by ion chamber and ferrous sulphate dosimetry and is reported to be accurate to $\pm$ 5%. The specimens were irradiated in air to the following dose levels:

Laminates 5×10^9, 10^{10}, 1.5×10^{10} rads.
Castings 10^9, 2×10^9, 5×10^9, 10^{10} rads.

4.2 The temperature of the specimens during irradiation would not be expected to rise significantly above the temperature of the shielding water (19°C).

*Instron Limited (Model TTCM) High Wycombe, Bucks.

5 RESULTS

Flexural strength and modulus values were derived from the load versus deflection curves; the mean values for the 5 replicates being given in tables 7 to 10 together with their standard deviations. The results are also presented graphically in figures 1 to 8.

6 DISCUSSION OF RESULTS

6.1 The use of large amounts of hardener in the MY 720/HY 906 resin system results in a worthwhile reduction in viscosity without significantly affecting the strength or radiation resistance of castings and laminates.

6.2 The incorporation of diglycidyl aniline as diluent in the MY 720/HY 906 system appears to have a slightly adverse effect on radiation resistance.

6.3 The use of small amounts of MY 720 as an accelerator for MY 740/HY 906 system appears to offer a slight improvement in radiation resistance over the tertiary amine accelerated system.

6.4 No significant differences in degradation were observed between the materials given different cure cycles.

7 CONCLUSIONS

Castings and glass cloth laminated specimens of selected epoxy resin systems have been evaluated for their potential use in high energy radiation environments. The order of radiation stability of the resin systems was similar for both cast and laminated specimens, but the cast specimens showed loss in strength at a much lower dose than the laminates.

With the cast systems, replication of mechanical test results was poor, the curves shown for these specimens were fitted by inspection and are therefore less reliable than those shown for the laminates, where replication was improved.

This report confirms the value of the tetraglycidyl amine resin, MY 720, in radiation resistant epoxy resin formulations. It is anticipated that further work may be undertaken in order to exploit the properties of this resin in room temperature curing systems.

8 REFERENCES

1. M J Price, R Sheldon RHEL/R105

2. R Sheldon, G B Stapleton RHEL/R152

3. J T Morgan, G Scott, R Sheldon, G B Stapleton RHEL/R196

4. D Evans, J T Morgan, R Sheldon, G B Stapleton RHEL/R200.

9 ACKNOWLEDGEMENTS

Acknowledgement is made to Mr P Bowles, Chief Engineer and to Mr G E Simmonds in whose department the work was carried out.

Resin	Hardener	Parts per 100 of Resin	Viscosity P at 24°C	Cure Cycle	Specimen No. Laminate	Specimen No. Casting
MY 720	HY 906	110	66	1	2	B
	HY 906	140	47	1	3	C
	HY 906	180	27	1	4	D
	HY 906 Diglycidylaniline	120) 20)	46	Laminate 1 Casting 7	5	E
MY 740	HY 906	80	32	-	-	-

Table 2 Use of Glycidyl Amine Resin as Accelerator

Resin	Hardener and Accelerator	Parts per 100 of Resin	Cure Cycle	Specimen No. Laminate	Specimen No. Casting
MY 790	HY 906 MY 720	100 5	2	9	H
MY 745	HY 906 MY 720	90 10	3	10	I

Table 3 Effect of Cure Temperature

Resin	Hardener	Parts per 100 of Resin	Cure Cycle	Specimen No. Laminate	Specimen No. Casting
LY 558	MY 906))) DY 062)	100 0.5	Laminate 1 Casting 7 4	6 7	F L
MY 720	HY 906	110	1 4	2 1	B A

Table 4 Other Hardener Systems

Resin	Hardener	Parts per 100 of Resin	Cure Cycle	Specimen No. Laminate
MY 790	HY 972	30	5	11
MY 750	Methyl Imidazole	3	6	12

Table 5 Cure Cycles

Cure Cycle No.	Cure Cycle		
1	4 hrs @ 100°C	3 hrs @ 150°C	4 hrs @ 200°C
2	4 hrs @ 130°C	4 hrs @ 150°C	
3		16 hrs @ 150°C	
4	24 hrs @ 100°C		
5	3 hrs @ 100°C	8 hrs @ 150°C	
6	3 hrs @ 70°C	6 hrs @ 150°C	
7	4 hrs @ 100°C	3 hrs @ 150°C	

Table 6 Conditions of Test

Specimen size	$\frac{1}{2}$" x 3" x $\frac{1}{8}$"
Span	2"
Rate of crosshead movement	0.1 cm/min
Chart speed	10 cm/min
Number of replicates	5

Table 7 Flexural Strength of Irradiated Laminates - Kgf/cm^2

System	Dose rads.			
	0	5×10^9	10^{10}	1.5×10^{10}
1	3147 (6.3%)	3251 (10.0%)	3013 (5.0%)	2536 (2.5%)
2	2899 (2.0%)	3131 (3.2%)	2956 (5.0%)	2535 (6%)
3	3040 (20.3%)	-	3108 (6.0%)	2400 (7%)
4	3184 (16.2%)	3168 (3.5%)	3160 (3.0%)	3231 (3.1%)
5	3044 (0.2%)	3352 (2.3%)	3283 (4.0%)	2384 (3.6%)
6	3600 (3.0%)	2761 (4.4%)	3012 (2%)	1930 (4%)
7	3423 (3.8%)	3306 (3.4%)	2935 (5%)	1821 (3%)
8	3002 (3.4%)	976 (10.2%)	861 (10%)	635 (9%)
9	3294 (16.4%)	3187 (14.7%)	2902 (2%)	1463 (5.7%)
10	3450 (5.9%)	2486 (7.5%)	2465 (3%)	1194 (13%)
11	3305 (5.9%)	2713 (3.3%)	1591 (3%)	1162 (2.9%)
12	3420 (12.3%)	3227 (3.7%)	2733 (10%)	1574 (4%)

Figures given are the mean values of 5 replicates together with standard deviations expressed as percentage of mean.

Table 8 Flexural Modulus of Irradiated Laminates - $Kgf/cm^2 \times 10^{-5}$

System	Dose rads.			
	0	5×10^9	10^{10}	1.5×10^{10}
1	1.53 (13%)	1.68 (17.4%)	1.62 (6%)	1.39 (10%)
2	1.53 (6%)	1.59 (6.8%)	1.54 (11%)	1.43 (7%)
3	1.87 (31%)	-	1.74 (1%)	1.43 (2%)
4	1.78 (7%)	1.68 (2.6%)	1.75 (5%)	2.01 (1%)
5	1.70 (3.1%)	1.69 (4.1%)	1.80 (6%)	1.54 (7%)
6	1.85 (6.4%)	1.70 (3.7%)	1.68 (5%)	1.58 (8%)
7	1.75 (4%)	1.75 (6.2%)	1.50 (6%)	1.46 (7%)
8	1.49 (2%)	0.92 (6.3%)	1.02 (1%)	0.99 (1.6%)
9	1.61 (21.2%)	1.73 (41%)	1.53 (6%)	1.34 (4.4%)
10	1.62 (4.3%)	1.53 (7.9%)	1.47 (6%)	1.23 (8.4%)
11	1.60 (9.4%)	1.51 (5.5%)	1.40 (14%)	1.53 (1.5%)
12	1.70 (14.5)	1.68 (7%)	1.74 (12%)	1.71 (6.9%)

Table 9 Flexural Strength of Castings - Kgf/cm^2

System	Dose rads.				
	0	10^9	2×10^9	5×10^9	10^{10}
A	998	934	1012	929	595
	(27%)	(3%)	(14%)	(13.1%)	(14%)
B	918	618	871	641	490
	(12%)	(35%)	(32%)	(41%)	(23%)
C	853	814	995	780	555
	(16%)	(8%)	(14%)	(12.4%)	(18%)
D	916	720	961	571	524
	(14%)	(25%)	(26%)	(36%)	(15%)
E	551		1087	-	471
	(44%)		(18%)		(14%)
F	1521	1033	1347	1008	859
	(5%)	(9%)	(11%)	(10.8%)	(6%)
H	1062		685	130	159
	(11%)		(12%)	(38%)	(7%)
I	1054	684	1040	239	192
	(8%)	(11%)	(29%)	(21%)	(9%)

Table 10 Flexural Modulus of Irradiation Castings $Kgf/cm^2 \times 10^4$

System	Dose rads.				
	0	10^9	2×10^9	5×10^9	10^{10}
A	3.64 (4%)	4.16 (7%)	4.35 (4%)	4.12 (1.6%)	4.21 (10%)
B	3.39 (5%)	3.85 (2%)	4.51 (10%)	4.05 (2.4%)	4.15 (1%)
C	4.19 (8%)	4.50 (1%)	4.45 (5%)	4.11 (0.7%)	4.25 (4%)
D	4.69 (5%)	4.71 (3%)	3.14 (34%)	4.19 (2.7%)	3.96 (8%)
E	3.83 (8%)	-	4.28 (3%)	-	4.48 (9%)
F	4.12 (4%)	4.20 (6%)	4.39 (5%)	4.09 (3.5%)	4.27 (6%)
H	4.64 (3%)	-	4.31 (5%)	4.13 (10.2%)	-
I	4.58 (8%)	3.97 (2%)	3.60 (14%)	3.86 (5.4%)	4.15 (5%)

Table 11 Glass to Resin Ratios for the Laminates

System	G/R Ratio (by wt.)
1	52:48
2	53:47
3	54:46
4	55:45
5	59:41
6	60:40
7	60:40
8	53:47
9	55:45
10	55:45
11	55:45
12	60:40

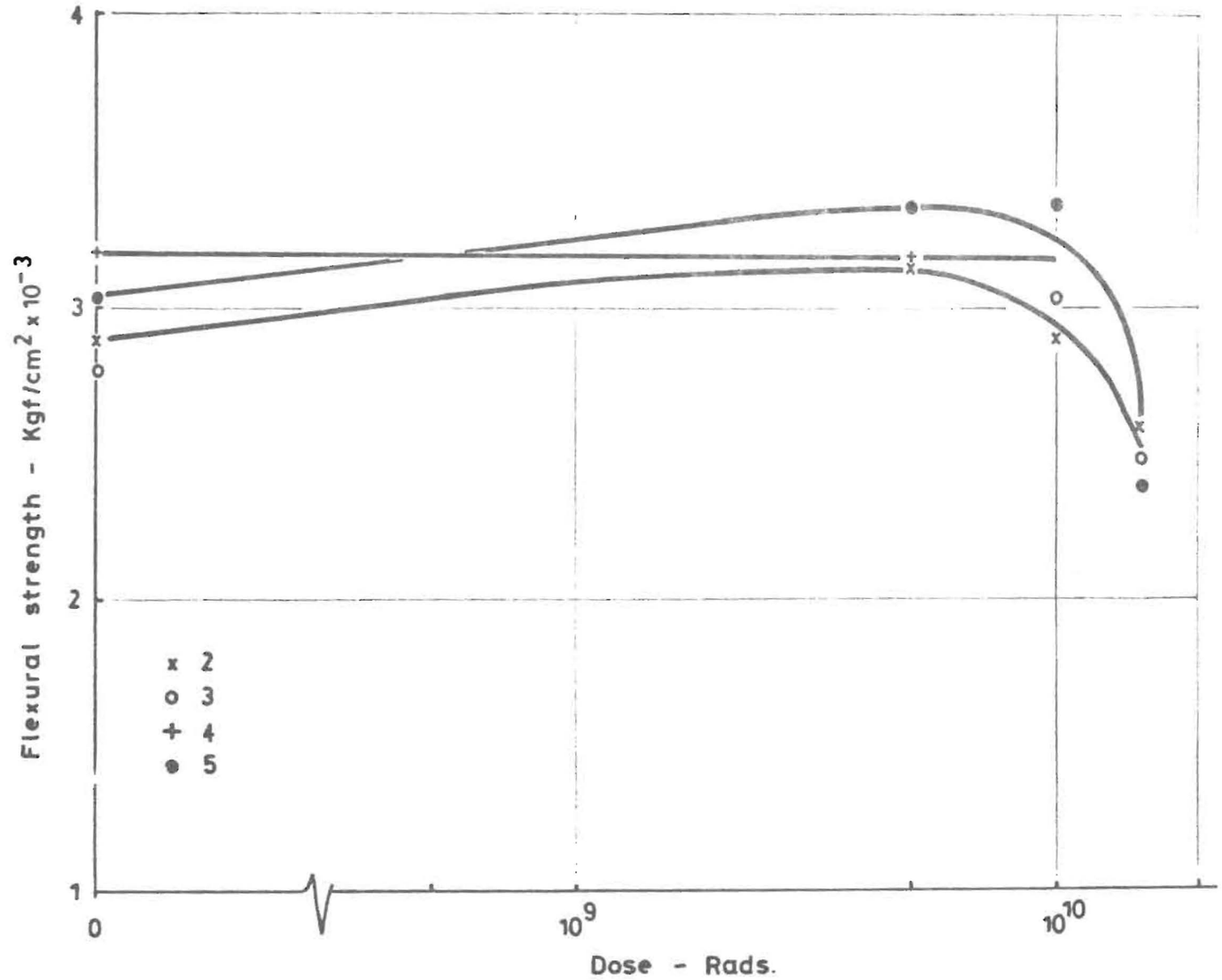

FIG. 1 LOW VISCOSITY MY 720 SYSTEMS. (LAMINATES).

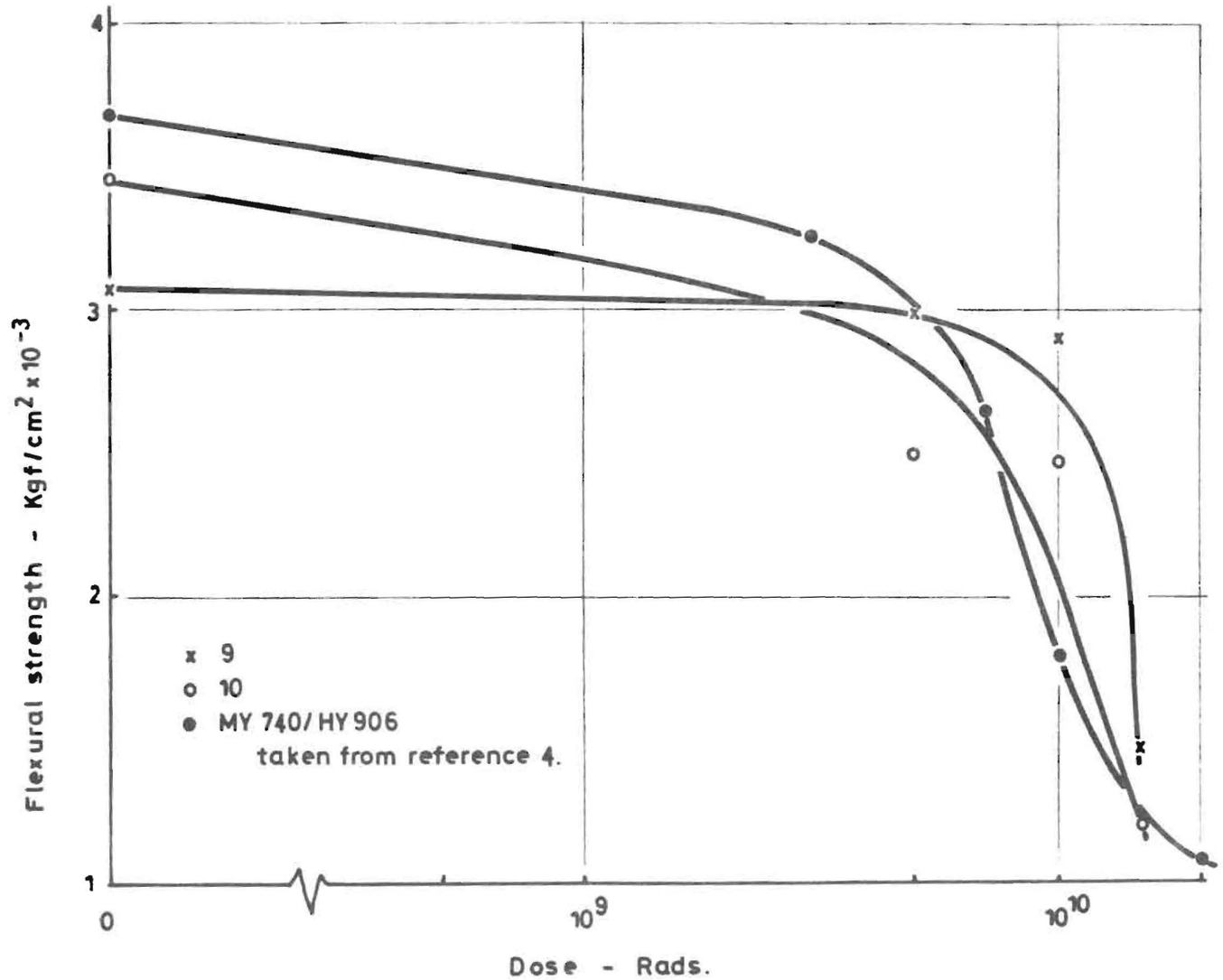

FIG. 2 GLYCIDYL AMINE ACCELERATED SYSTEMS. (LAMINATES).

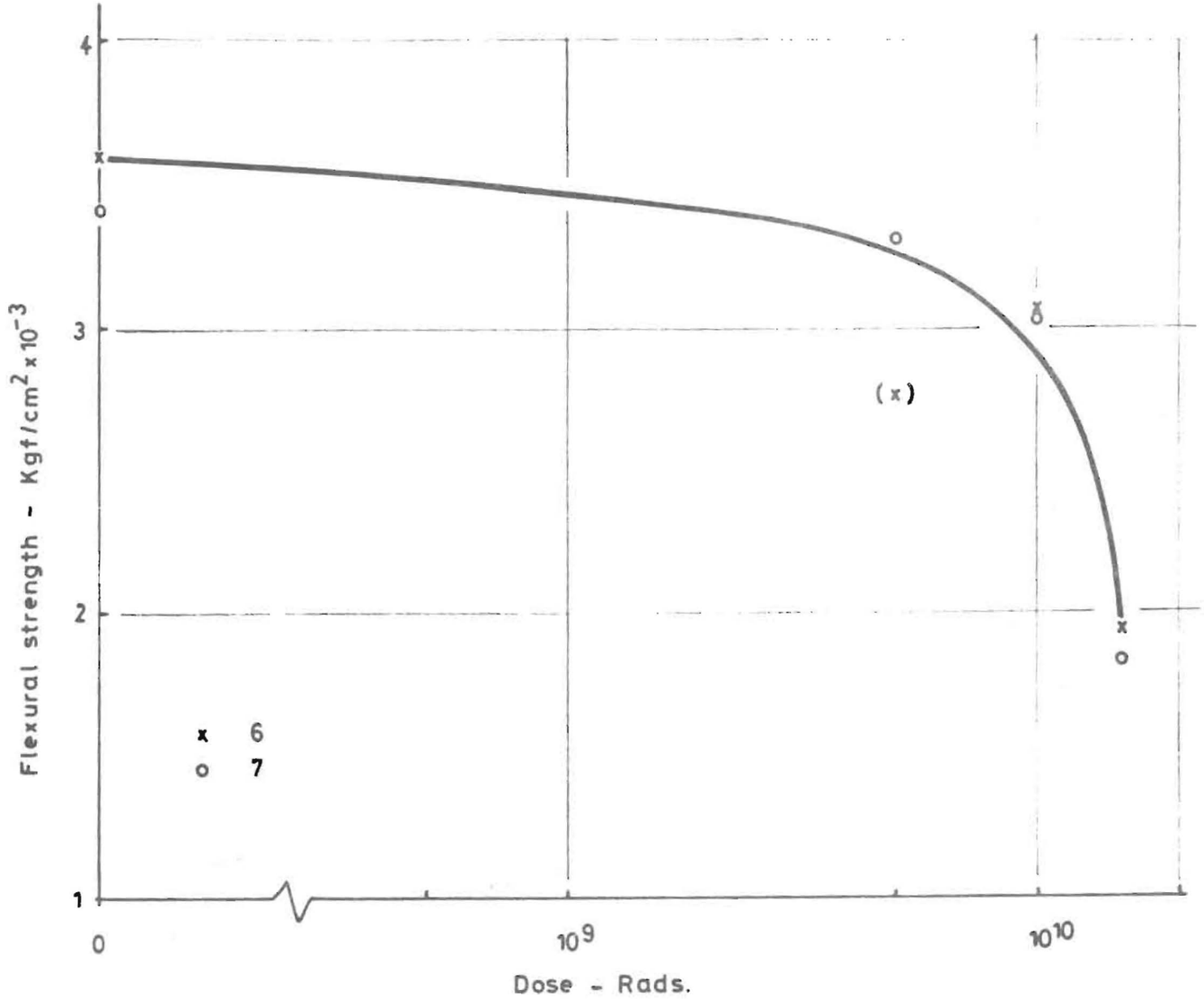

FIG. 3 EFFECT OF CURE TEMPERATURE ON LY 558 / HY 906 (LAMINATES).

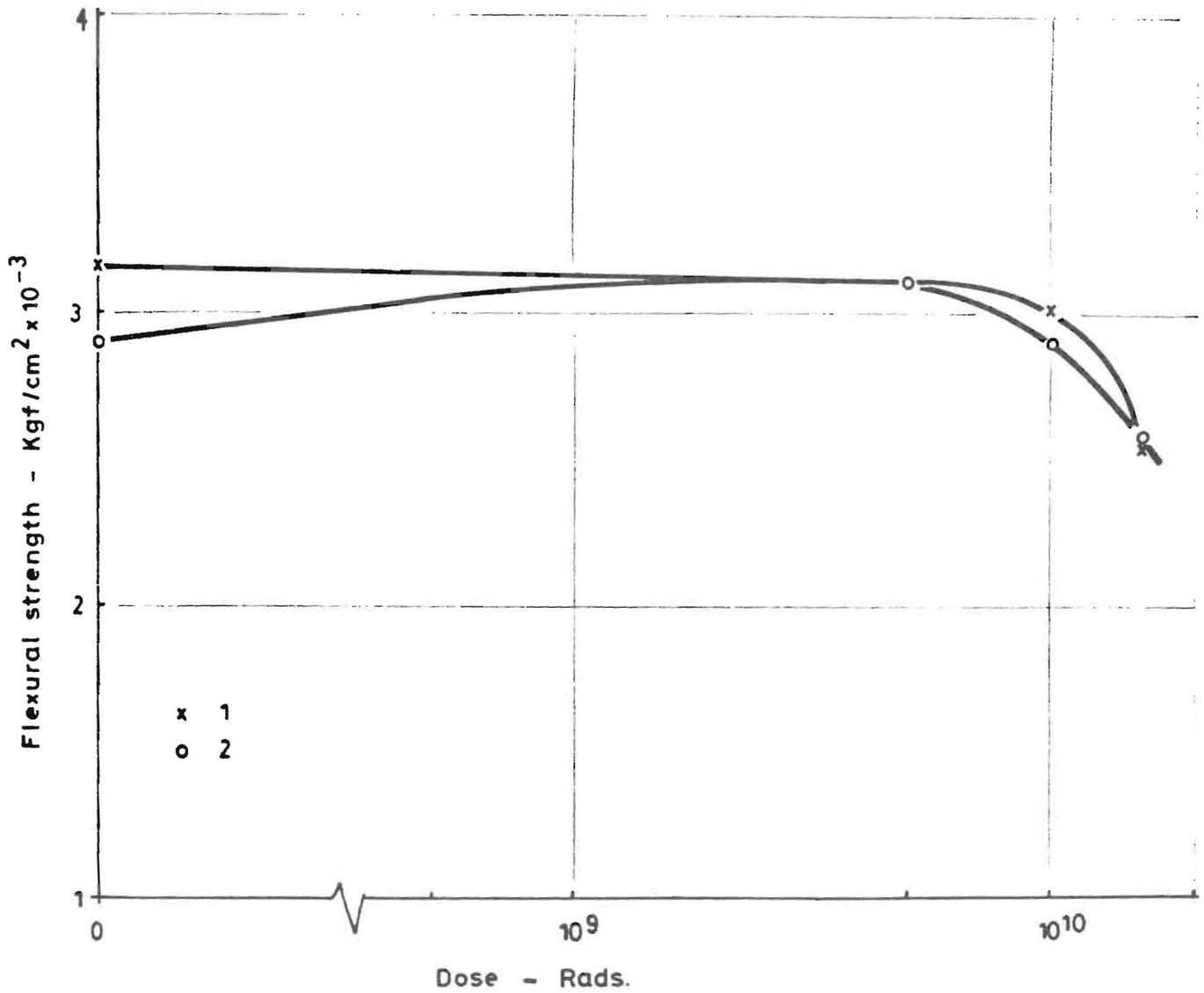

FIG. 4 EFFECT OF CURE TEMPERATURE MY 720/ HY 906 (LAMINATES).

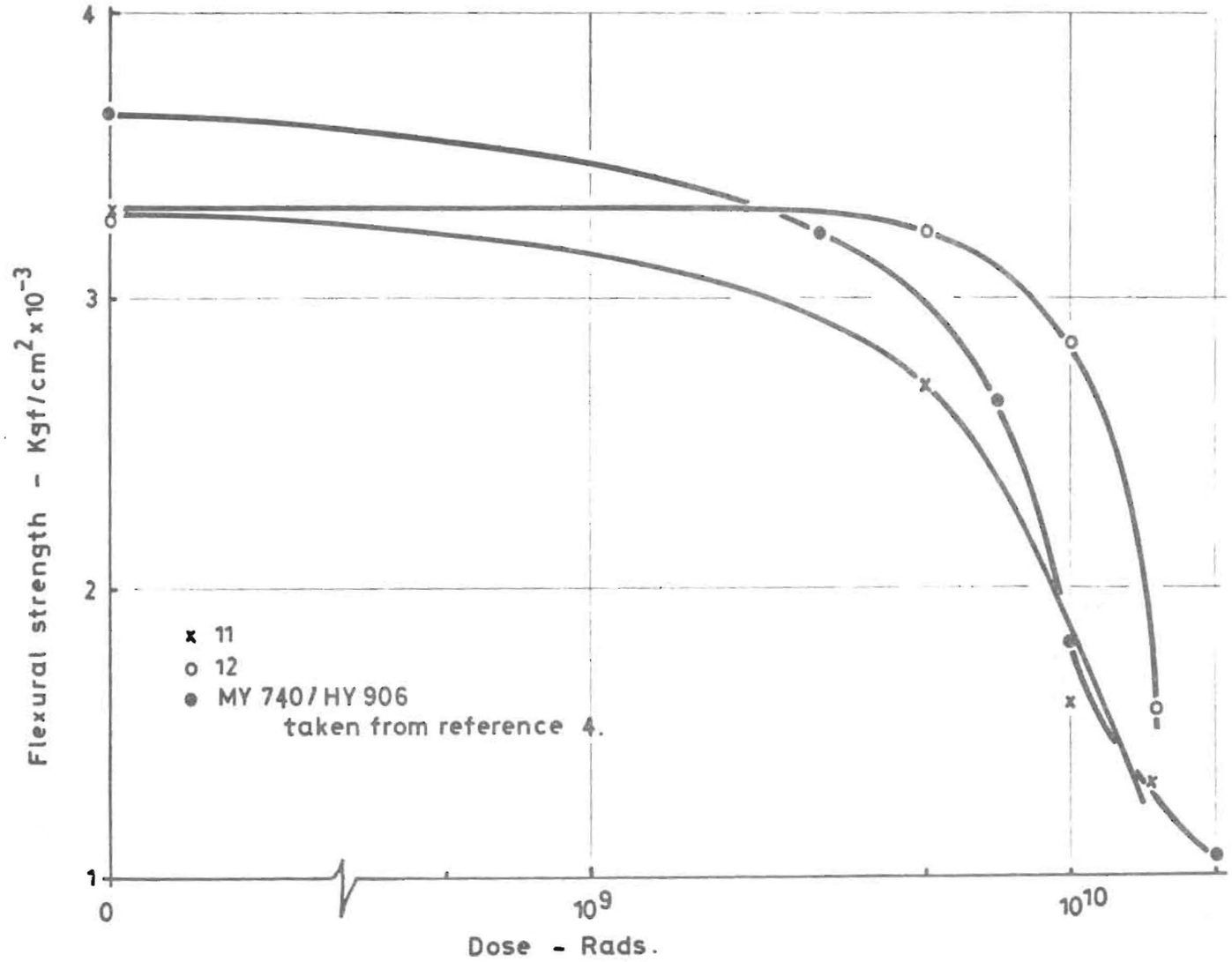

FIG. 5 COMPARISON OF 3 DIFFERENT CURING AGENTS (LAMINATES.)

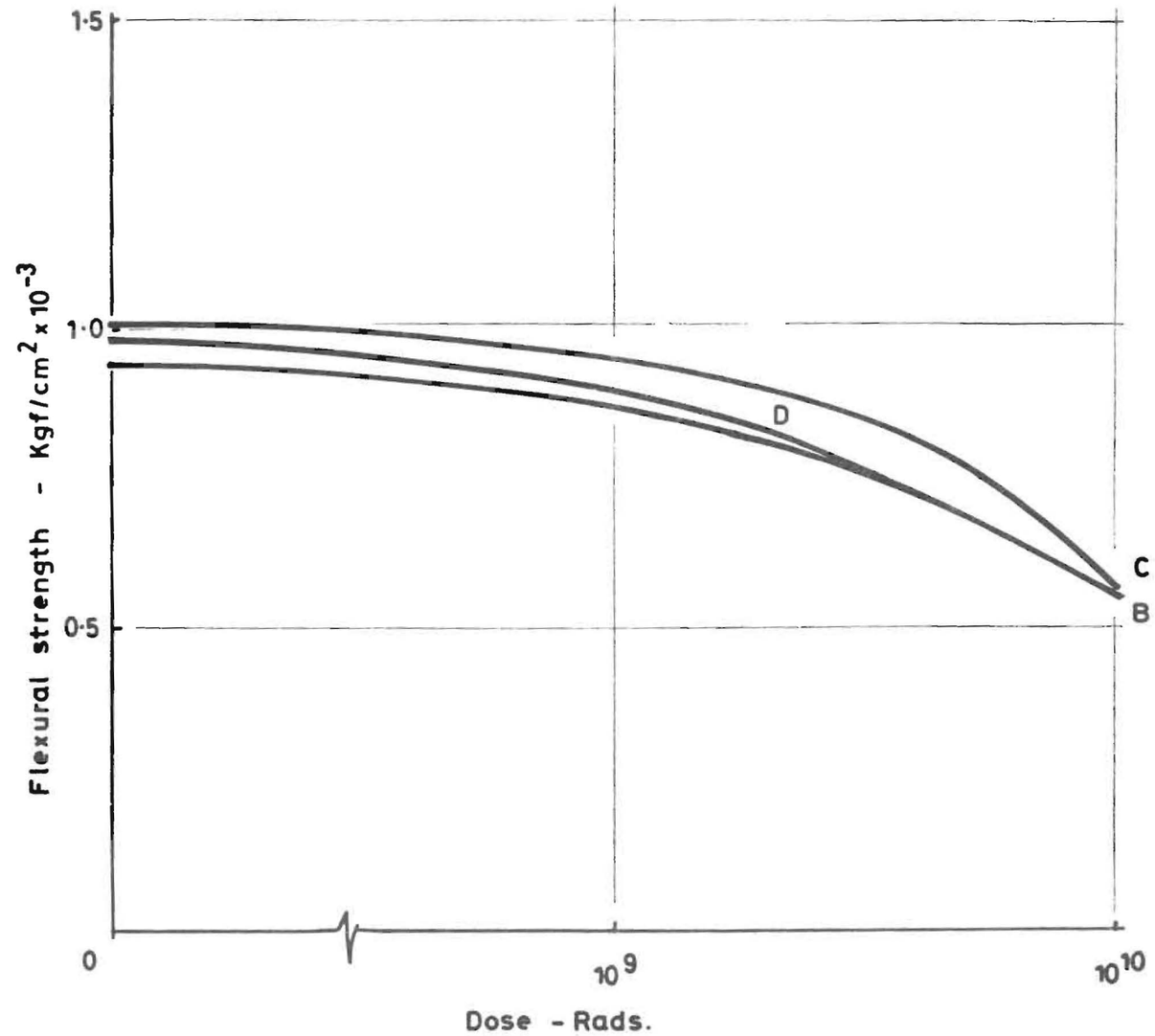

FIG. 6 LOW VISCOSITY MY 720 SYSTEM (CASTINGS).

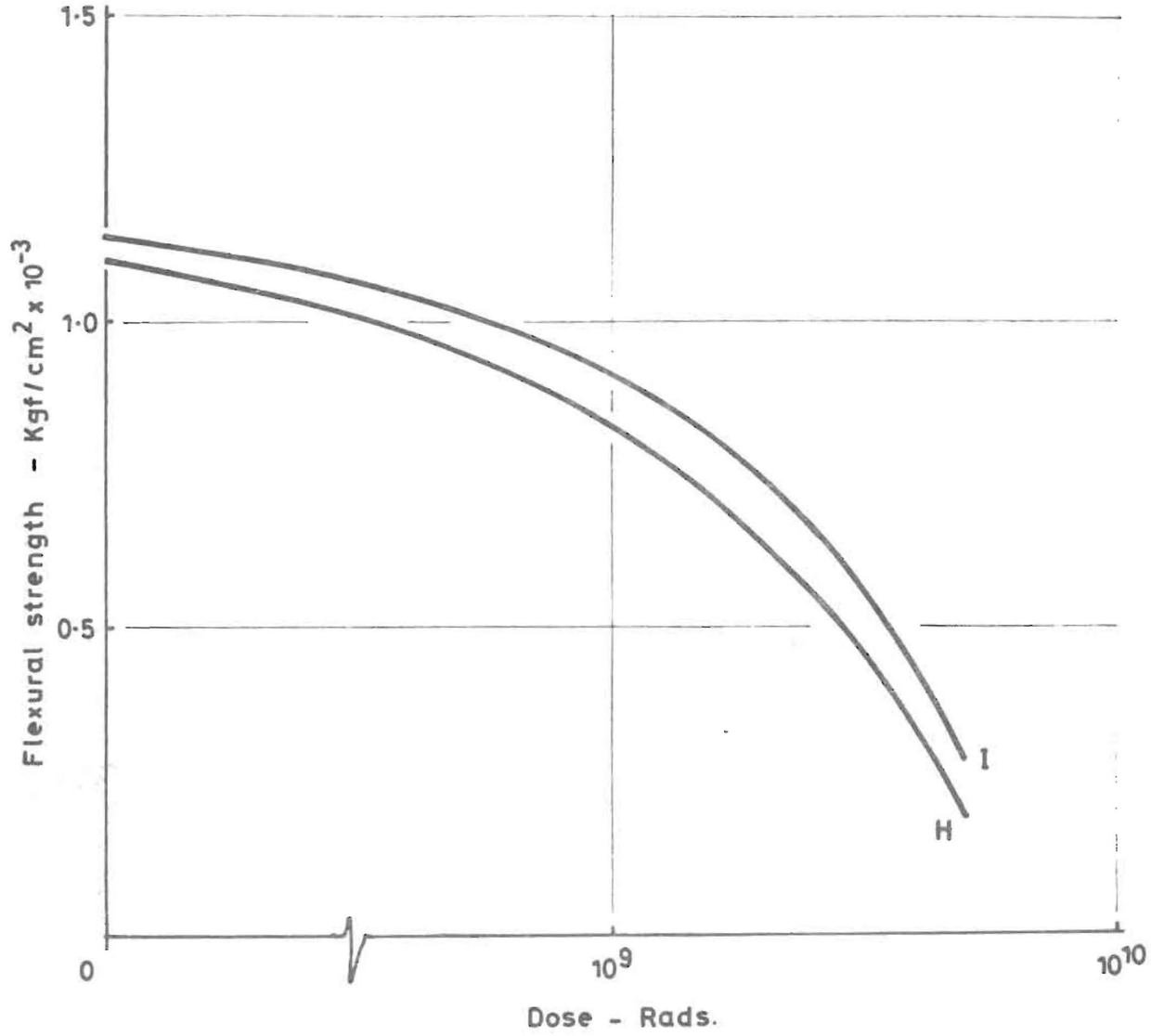

FIG. 7 GLYCIDYL AMINE ACCELERATED SYSTEMS (CASTINGS).

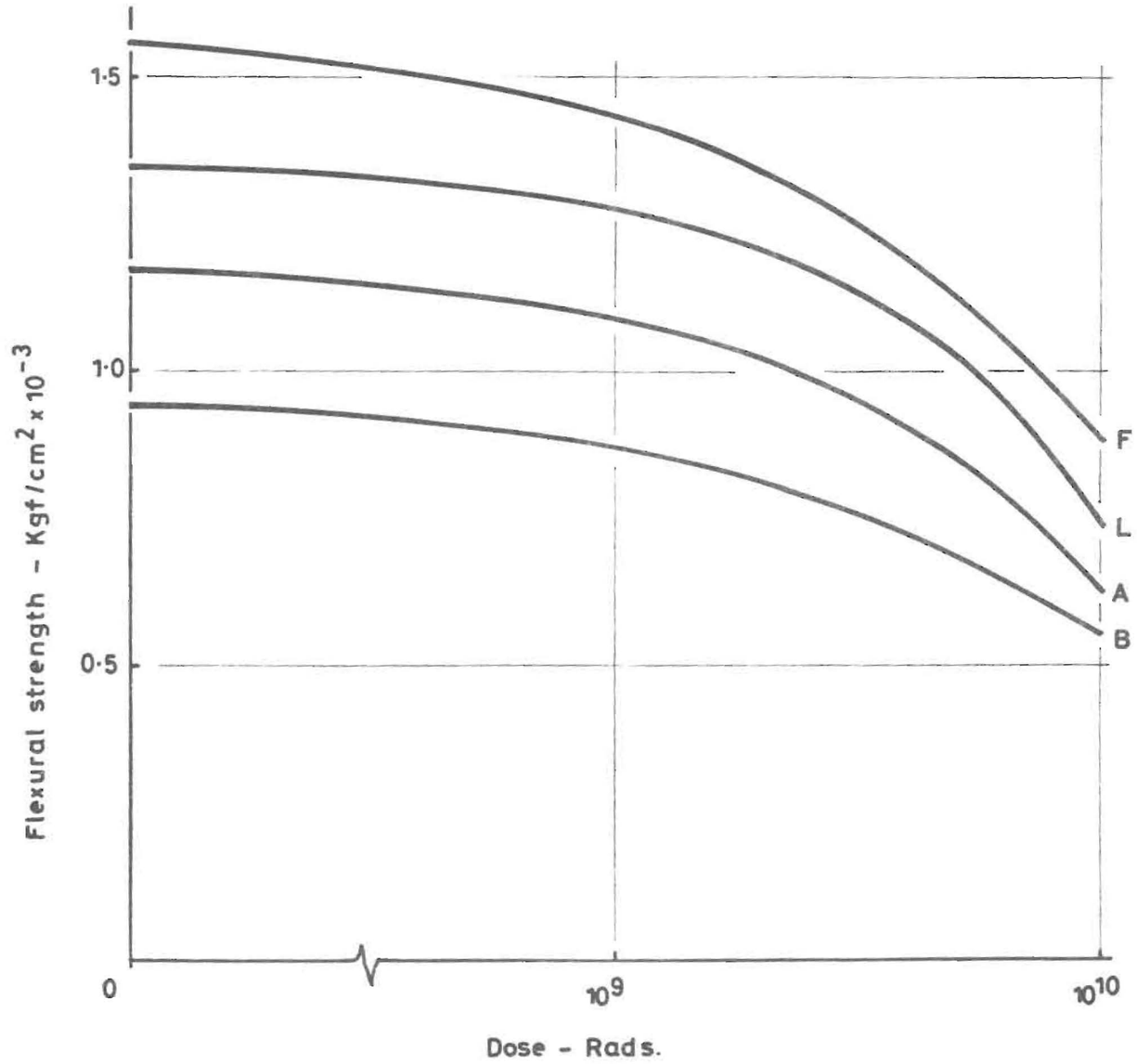

FIG. 8 EFFECT OF CURE TEMPERATURE. (CASTINGS).