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"CARILON" POLYMERS FOR AUTOMOTIVE USE
A comparative study
(July - November 1989)

by

R.E.C. BARTON and J.C.M. JORDAAN

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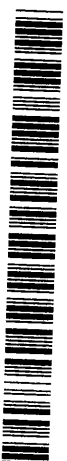


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Approved by: A.A. Broekhuis

SUMMARY
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One of the envisaged applications for CARILON EP polymers is exterior automotive body panels. To assess its suitability for this type of application a comparative study with more established plastic materials in this field has been undertaken. A CARILON EP compound developed for this purpose by Westhollow Research Centre in Houston, has been compared with the thermoplastic market leader, Noryl GTX, a Nylon/PPO blend from General Electric Plastics and a sheet moulding compound (SMC), a random glass fibre thermoset polyester system, in the form of a commercially available Citroen BX bonnet.

The overall performance of the SMC system was typical for a glass reinforced chemically cross-linked matrix. The parts are very stiff and retain a high level of stiffness at elevated temperatures. The coefficient of linear thermal expansion (CLTE) is low and of the same order of magnitude as steel. In addition the material has no tensile elongation and good creep performance.

Comparing the two thermoplastic materials, the Noryl GTX 910 exhibited a marginally better overall balance of properties. It was less sensitive to temperature in all the thermal performance related properties evaluated, but more sensitive to water than CARILON. The impact performance of this CARILON compound was inadequate, yielding brittle failures even at ambient temperature. Reformulation to improve this deficiency will be necessary as the automotive industry is seeking materials that give ductile failures at temperatures below 0°C.

It may be concluded that CARILON can become a potential competitor to Noryl GTX in this application, especially as product quality has improved significantly since this evaluation and all compounding modification possibilities as yet have not been fully explored. Both thermoplastic materials on the other hand would have difficulty competing technically with a thermosetting system such as SMC, and would require the development of non-conventional methods of incorporating glass fibre networks in the plastic matrix.

Future work will be directed at reformulation to improve the impact resistance. A full evaluation of available mineral fillers and short glass fibres which can be compounded with CARILON and used on conventional injection moulding machines will be carried out.

August 1990

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"CARILON" POLYMERS FOR AUTOMOTIVE USE

A Comparative study

1. INTRODUCTION

The automotive industry employs a variety of plastic materials for exterior parts such as bumpers, viz :PU-RIM, Polyurea, EPDM/PP, PC/PBT, amorphous Nylon, TPE/PC, PPO/Nylon (1)(2)(3). More specific, for body panels the materials list is much shorter. It consists almost exclusively of thermosets i.e. glass reinforced polyester systems in various production technology forms, sheet moulding compound (SMC, BMC, ZMC) and glass reinforced PU-RIM. The main reason for this is that these parts must be able to withstand paint line stoving temperatures and must have a thermal expansion performance as close to steel as possible.

At present there is no thermoplastic polymeric material available that can meet all industry's requirements. Industry however would prefer to use these materials as they offer good production economics. This coupled with the availability of a number of new polymer alloys which offer higher elevated temperature dimensional stability, has generated a considerable amount of development effort into the possible application of thermoplastics in fenders. Fenders were chosen as a development target because higher CLTE's and lower stiffness can be tolerated for vertical parts.

This end-use area has also been identified as a potential outlet for CARILON polymer, where it's high temperature performance and the option to regulate and control it within a certain temperature range, could be advantageous. WRC have demonstrated that large parts could be moulded and painted, distortion free from CARILON polymer (4).

In order to be able to define clear development directives this comparative evaluation exercise was initiated. The CARILON EP based compound as developed by WRC for this purpose has been compared to the thermoplastic market leader, Noryl GTX 910, a Nylon/PPO blend and SMC, in the form of a commercially available Citroen BX bonnet. A similar comparative exercise has been carried out in WRC without the inclusion of SMC (5). As the European industry requirements are likely to be different to the US as cars are constructed differently, it was felt desirable to carry out this exercise on materials that at present are used or under study in the European auto-industry.

2. EXPERIMENTAL

2.1. Materials Used

- a) The WRC fender compound had the following composition : (1)
- | | |
|------------------------------|-----------|
| CARILON EP mixed batches (2) | 84.5 % wt |
| Fibreglass OCF 492 AA | 8.0 % wt |
| Primacore 1430 | 5.0 % wt |
| EVA grey pigment | 2.5 % wt |
- b) Noryl GTX 910 a Nylon 6/PPO blend ex General Electric Plastics with PPO as the dispersed phase.
- c) Polyester based thermoset system - sheet moulding compound (SMC) in the form of a purchased Citroen BX bonnet.

Notes: 1) This compound was mixed and extruded by Paragon Development Corporation, Connecticut U.S.A.
2) Average LVN 1.7 dl/g.

2.2. Test Methods Selected

- a) Rheological behaviour, complex viscosities as a function of frequency by cone and plate rheometry.
- b) Tensile properties ASTM D638M (type 3). Measured at 23°C and after one week in air.
- c) Flexural modulus/strength ASTM D790. Span to depth 16:1. Span 100mm for CARILON and Noryl systems and 50 mm for the SMC. Test temperature -30/23/60/120°C. Also at 23°C after one week's immersion in water.
- d) Notched Izod impact ASTM D256 "A" notch. Test temperature -30°C and 23°C.
- e) Rheometrics impact. 4.4m/sec at 23 and -30°C.
- f) Heat distortion temperature ASTM D648-82. 1.82 MPa load.
- g) Heat sag ASTM D3769-85. 6" overhang, one hour at 160°C.
- h) Shrinkage after one hour at 180°C.
- i) Hardness Shore D scale.
- j) Water pick-up ASTM D570-81.
- k) CLTE between -50 and 100°C.
- l) Creep strain at 23°C. Using 10 and 20 MPa stress loadings.

2.3. Characteristics of the Citroen BX Bonnet

Size : 1.0 X 1.35 m
Weight : 10.3 kg
Thickness: 3.0 mm. Exept towards the front lip where the thickness gradually increased from the outside edge to the centre (3.0 - 5.0 mm).

Design of the reinforcing ribs: Depth 20 mm, taper 3.0 to 1.75 mm.

Hinge attachment: Via 3 bolts screwed into the SMC after manufacture.

Bonnet locking assembly: Via 5 bolts screwed into the SMC after manufacture.

Ejector points: This appears to be via 5 large pins situated in the front lip and 90 small pins situated in the reinforcing rib system.

Paint system: Consists of three coats:

- 1) Black priming layer
- 2) Yellow filling coat
- 3) Coloured topcoat

Total film thickness 0.075 mm. Adhesion of the paint film to the SMC was assessed by cross hatch adhesion test, which was

Glass content: 50% wt. (without the paint layer) determined by ashing at 900°C for two hours. Samples were cut from three different places in the bonnet.

Appearance of the glass, after ashing, suggests it is in the form of a random mat. When pieces of bonnet were ashed containing reinforcing ribs it was clear there was no connection in the glass layers in the flat areas of the bonnet and the ribs, implying that the ribs were filled by using a separately made preform. After ashing there was also present in the glass a grey filler, which is probably the remnants of pigment and magnesium oxide thickener used in the polyester resin.

2.4. Sample preparation

- a) For rheological behaviour. This test was only carried out on the CARILON and Noryl systems; 1 mm thick plates were compression moulded from nibs using a schedule of 2 min at 245 and 285°C respectively, at a pressure of 10 T. From these plates 25 mm diameter discs were cut for the test.
- b) CLTE, for the SMC, small samples were cut from the bonnet. For the CARILON and Noryl systems 3 mm thick samples were prepared from compression moulded plates. The schedule used was the same as that given in a) above except that the time used was 4 mins.
- c) For all other tests, CARILON and Noryl samples were prepared by injection moulding; details of the conditions used are given in Table 1. The SMC samples were prepared by cutting them from the flattest part of the bonnet; samples were cut in two directions to check the degree of anisotropy.

Results of the physical property determinations are given Table 2 - 8 and Figure 1 - 8.

3. RESULTS AND DISCUSSION

3.1. Rheological Behaviour

Complex viscosity measurements were determined at 240°C for the CARILON material, and 290°C for the Noryl GTX. A frequency of 0.5 - 500 rad/sec was used, which for polymers generally coincides numerically with the shear rate. Measurements were made after 1, 10 and 22.5 min. The results are shown graphically in Figures 2 - 4.

The greater stability of the Noryl is clearly shown in Figure 2, where the curves determined after 1 min and 22.5 min show little change whereas the curves for CARILON indicate a viscosity increase with time and this despite a 50°C difference in test temperature. The same pattern is repeated for the dynamic shear modulus, this relatively sharper increase of elastic modulus is an indication of crosslinking taking place. A comparison of both results for the CARILON compound is given in Figure 3 and for Noryl GTX in Figure 4.

3.2. Thermal Expansion Coefficient

The CARILON compound and the Noryl GTX 910 were considered to be isotropic (expansion the same in all directions) because sample preparation was carried out by compression moulding, so were measured in the Z direction. The thermal expansion of the clearly non-isotropic SMC sample was measured in all three directions. The dimensional changes of a polymer sample during heating or cooling are often determined by both the expansion coefficient and the thermal history of the sample. Figure 5 shows the length change of the CARILON compound as a function of the temperature during the first heating scan followed by a cooling scan. Relaxation of frozen-in compression forces results in a sample length increase (in the Z direction) after a heating scan to 100°C. The results of the second and third heating scans were nearly identical. The obtained expansion coefficient data were now therefore typical material values. Both thermoplastic materials showed nearly identical thermal history effects. The length of the polyester sample however, decreased due to the thermal treatment during the first heating scan, Fig 6 ; this shrinkage was probably due to a post cure effect. The length/temperature relation of these samples was therefore measured during two subsequent heating scans. Fig 7 shows the length increase of the three investigated systems during the first heating scan from -40°C up to 100°C. The high glass loading of the polyester system resulted in a very small change in length (about 0.4%). The length change of the Noryl system was clearly smaller than that of the CARILON based compound. These differences were reflected in the linear expansion coefficient values see Figs 8 and 9. The step-wise change of the CLTE of both thermoplastic systems was caused by the relaxation of internal stresses, this became possible after passing the glass/rubber transition temperature. During the second heating scan a more or less continuous CLTE was exhibited, the usually measured behaviour of semi-crystalline polymers. The non-isotropic behaviour of the glassfibre reinforced polyester system is shown in Figure 10. The glassfibres are randomly distributed in the XY plane (no significant differences in expansion in the XY plane). As all fibres are distributed at right angles to the vertical plane, resin expansion was therefore possible in the vertical plane thus giving a slightly higher expansion in this direction.

3.3. Other Physical Properties

The physical test results obtained reflected the nature of the individual materials. The essentially semi-crystalline nature of the Noryl, nylon modified with an amorphous polymer, PPO, meant that mechanical properties decreased at a modest rate over the temperature range tested, (max. 120°C), with no large drop-off at the Tg as it was not reached (135°C for PPO). An illustration of this can be seen in Table 2 which shows the variation in flexural modulus with temperature. CARILON on the other hand (semi-crystalline polymer) has a Tg of ca 20°C; the effect of this meant properties changed at a rapid rate around this temperature, this was true for both flexural and impact properties. Similarly the effect on impact properties was less marked for the Noryl system, although still considerable, a 50% loss being experienced when tested at -30°C compared to 23°C. This was true for both notched impact, Table 5, and Rheometrics impact, Table 9. This latter test showed the marked superiority of both the Noryl and SMC systems both giving ductile failures at 23°C, whereas the CARILON compound gave only brittle failures. It was interesting to note that the Noryl and SMC systems gave about the same failure energy at 23°C, a surprising result bearing in mind the large percentage of glass in the SMC system. The polyester based SMC system performed typically for a glass filled chemically cross-linked system. The product was much stiffer and retained its stiffness better at elevated temperatures; it had a low CLTE, of the same order of magnitude as steel, high heat distortion temperature, negligible heat sag, low tensile creep and no reduction in tensile properties after heat ageing. The results demonstrate why the automotive industry has had the confidence to commercially utilise this material for exterior body parts. Both the thermoplastics were significantly inferior in all properties tested.

In general it can be concluded that potentially CARILON could become a serious competitor for Noryl GTX in this field of application, especially taking into account the product quality improvements achieved by manufacturing, since this evaluation took place. At the same time the two thermoplastics would have difficulty competing with a glass filled thermoset such as SMC and would require non-conventional methods of glass incorporation whilst retaining thermoplastic production economics. Effort in this area may become necessary if suppliers of suitable thermoplastics want to participate in this future market because glass filled RIM polyurea systems are developing into an attractive alternative; these systems combine thermoplastic production economics with glass filled thermoset properties

4. CONCLUSIONS

- 1) SMC is superior to Noryl and CARILON in all properties tested, the glass filler and thermoset matrix render this compound very stiff; it shows only small changes in physical properties over the temperature range tested. Heat distortion is high, $>280^{\circ}\text{C}$, it has no heat sag. Measurable CLTE occurs only in the Z plane as the material is non-isotropic; creep measured at a stress loading of 20 MPa, twice that used for the two thermoplastic compounds, gave a strain of only 0.2% after 900 hours, against 1.1 - 1.3% for the former materials.
- 2) Comparing the two thermoplastic materials, Noryl and CARILON, Noryl has marginally better properties especially where resistance to heat is concerned. It has a higher HDT, and tensile properties are less affected after one week at 120°C , also CLTE is lower.
- 3) Noryl possesses a more uniform stiffness response to a temperature change range ($-30^{\circ} - +120^{\circ}\text{C}$) than CARILON, which shows a sharp increase in stiffness below its T_g of ca 20°C .
- 4) The CARILON compound shows rather poor impact properties, compared to Noryl, in both notched Izod and Rheometrics tests. Since this work was completed we know that current CARILON production has much better impact properties and is now as good if not better than Noryl.
- 5) CARILON is the most hydrophilic of the three systems tested and SMC is the worst; the latter is probably due to the "wicking" effect of the cut glass fibres created when the test samples were prepared.
- 6) Creep under 10 MPa stress loading for 900 hours is slightly better for Noryl than CARILON, but both have $< 1.5\%$ strain at this time.
- 7) Dynamic viscosity measurements clearly show that over the frequency and temperature range tested, Noryl is more stable than CARILON.

Amsterdam, August 1990

TABLE 1
INJECTION MOULDING CONDITIONS

System	Barrel Temps				Inj P bar	Inj speed %	Dosing dist mm	After P bar	Cool time s
	T1	T2	T3	T4					
CARILON Disc	220	235	235	245	70	70	47.5	50	17
CARILON flex bar	220	235	235	245	90	70	52	50	17
CARILON tensile	220	235	235	245	85	70	52.5	44	17
Noryl disc	251	292	291	263	70	100	33	25	12
Noryl flex bar	280	295	295	290	65	10	49.5	25	9
Noryl tensile	280	295	295	290	65	70	51	31	17

Note T1 temp of hopper.

T4 temp of nozzle

Table 2
Flexural Properties

	Test temperature							
	-30°C		+23°C		+60°C		+120°C	
	Mod GPa	Strength MPa	Mod GPa	Strength MPa	Mod GPa	Strength MPa	Mod GPa	Strength MPa
CARILON	4.83	107	1.59	42.7	1.0	29.4	0.48	17.3
Noryl	2.58	99	1.69	61.6	1.23	42.5	0.87	30.2
SMC Trans	12.6	176	11.1	148	8.2	133	6.0	106
SMC Parallel	13.1	153	11.3	137	8.1	120	5.1	75

Note: Flexural strength figures are real for the SMC system but are calculated at 5% strain for the CARILON and Noryl systems.

Table 3
Flexural Properties After 1 Week's Immersion in
Water at 23°C

	Modulus GPa	Stress at 5% Strain MPa	Water Pick-up %wt
CARILON	1.47 (-7%)	41.2 (-3.5%)	0.6
Noryl	1.36 (-20%)	48.4 (-21%)	0.8
SMC Trans	8.95 (-20%)	119.3 (-20%)	0.9

Table 4
Tensile Properties

	Tensile strength MPa (max)		Elongation %	
	As made	After 1 week at 120°C	As made	After 1 week at 120°C
CARILON	46.6	61.0 (+30%)	215	22 (-90%)
Noryl	55.0	60.8 (+11%)	115	42 (-63%)
SMC trans	62.3	65.9 (+5.8%)	0.75	1.4
SMC parallel	64.5	58.6 (-10%)	0.9	1.0

Figures in brackets represent percentage change in the property.

Table 5
Notched Izod Impact

	Impact at 23°C		Impact at -30°C	
	KJ/M2	J/M	KJ/M2	J/M
CARILON	18.1	185	4.8	49
Noryl	20.7	212	9.9	101
SMC trans	98	998	94.5	964
SMC parallel	82	845	---	---

Table 6
Shrinkage %

After heating moulded samples for 1 hr at 180 C

	Dumbbell			Flex mod bar		
	Length	Width	Thickness	Length	Width	Thickness
CARILON	0.77	0.25	0	0.44	0	0
Noryl	1.0	0.25	0	0.7	0	0
SMC trans and parallel	0	0	0	0	0	0

Table 7
Water Pick-up

		Time in days % wt increase			
		1	2	6	35
CARILON	(1)	0.42	0.59	1.01	2.1
	(2)	0.42	0.59	1.02	2.1
Noryl	(1)	0.58	0.81	1.38	3.1
	(2)	0.61	0.86	1.43	3.1
SMC plus	(1)	1.34	1.51	2.55	4.0
paint	(2)	1.29	1.53	2.55	4.1
SMC minus	(1)	1.39	1.69	3.01	4.3
paint	(2)	1.64	1.97	3.33	5.0

Table 8

Miscellaneous Properties

HEAT DISTORTION TEMPERATURE 1.8 KPa LOAD

CARILON	106 C
Noryl GTX 910	152.5 C
SMC system	>280 C

HEAT SAG 1Hr AT 165 C, 6" OVERHANG

CARILON	2.0 mm
Noryl GTX 910	Not measured
SMC system	0.15 mm

<u>HARDNESS</u>	<u>SHORE D SCALE</u>	
	<u>Initial</u>	<u>After 30 sec</u>
CARILON	80	76
Noryl GTX 910	79	75
SMC	90	88
SMC (no paint)	89	87

TABLE 9

RHEOMETRICS IMPACT RESULTS

Measurements carried out at 23 and -30 C using a piston speed of 4.4 M/sec. Piston diameter 15.7mm.

System	Test Temp C	Yield Force N	Yield Disp. mm	Yield Energy J	Total Energy J	Total Disp mm	Type of Failure
CARILON	23 -30	1770 Not Tested	6.6	2.9	3.7	7.4	Brittle
Noryl	23 -30	4630 2870	14 7.5	29.0 6.3	38.8 13.1	17 8.6	Ductile Br/D
SMC	23 -30	3600 4030	6.6 5.1	9.3 8.7	24.8 23.1	14 13.8	Ductile Ductile

Br/D = Brittle/Ductile failure

FIGURE 1
Flexural strength properties

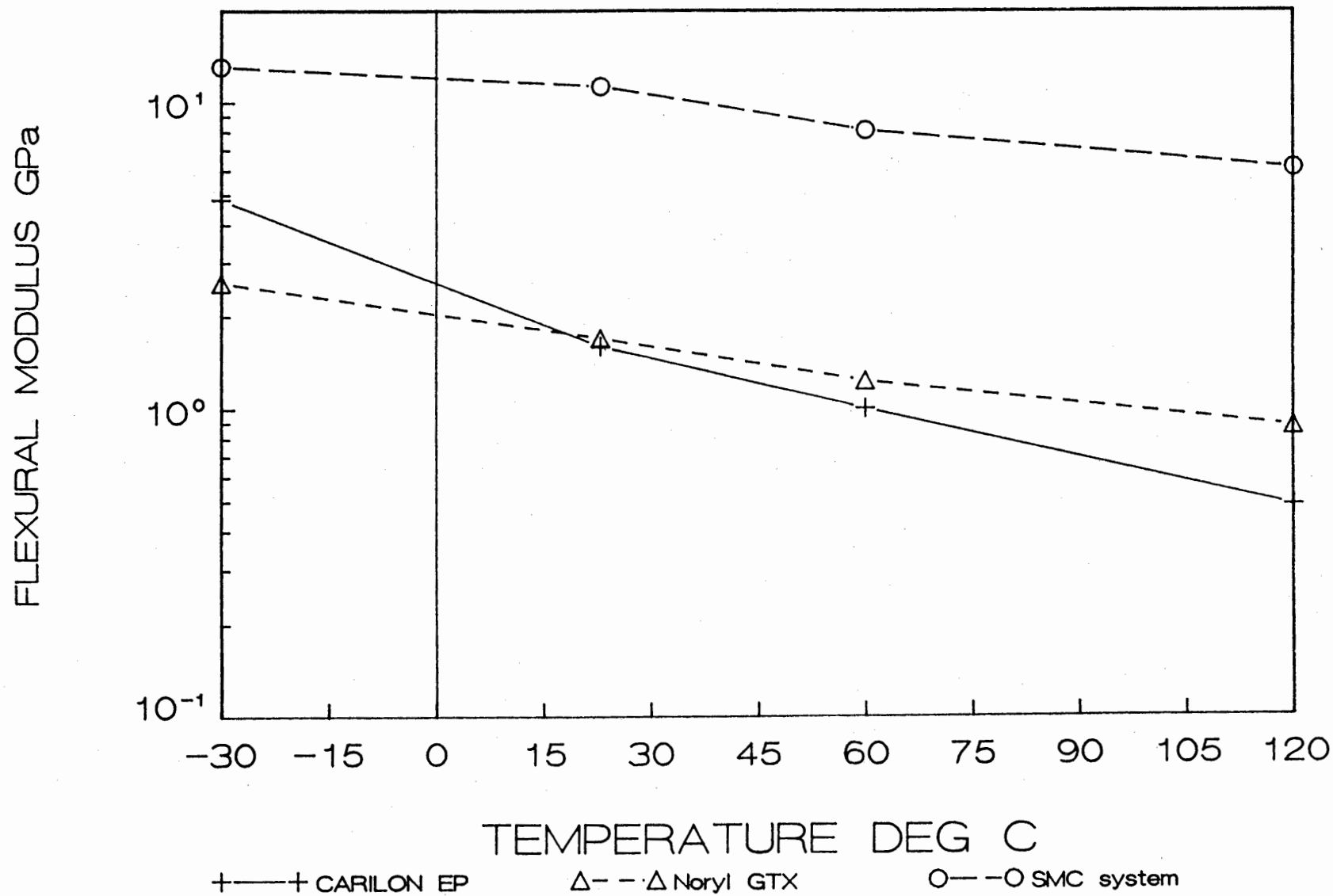


Fig. 2 DYNAMIC VISCOSITY
 "CARILON" FENDER COMPOUND vs NORYL GTX

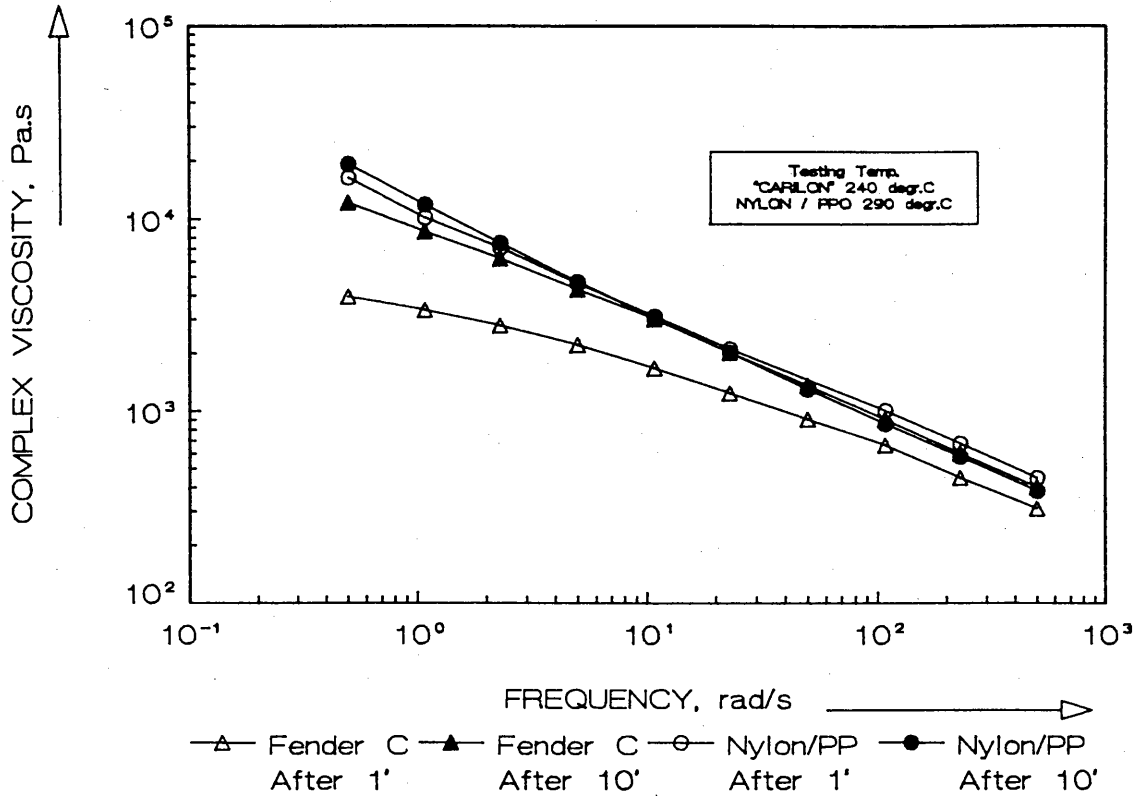


Fig. 3 DYNAMIC VISCOSITY
 "CARILON" FENDER COMPOUND

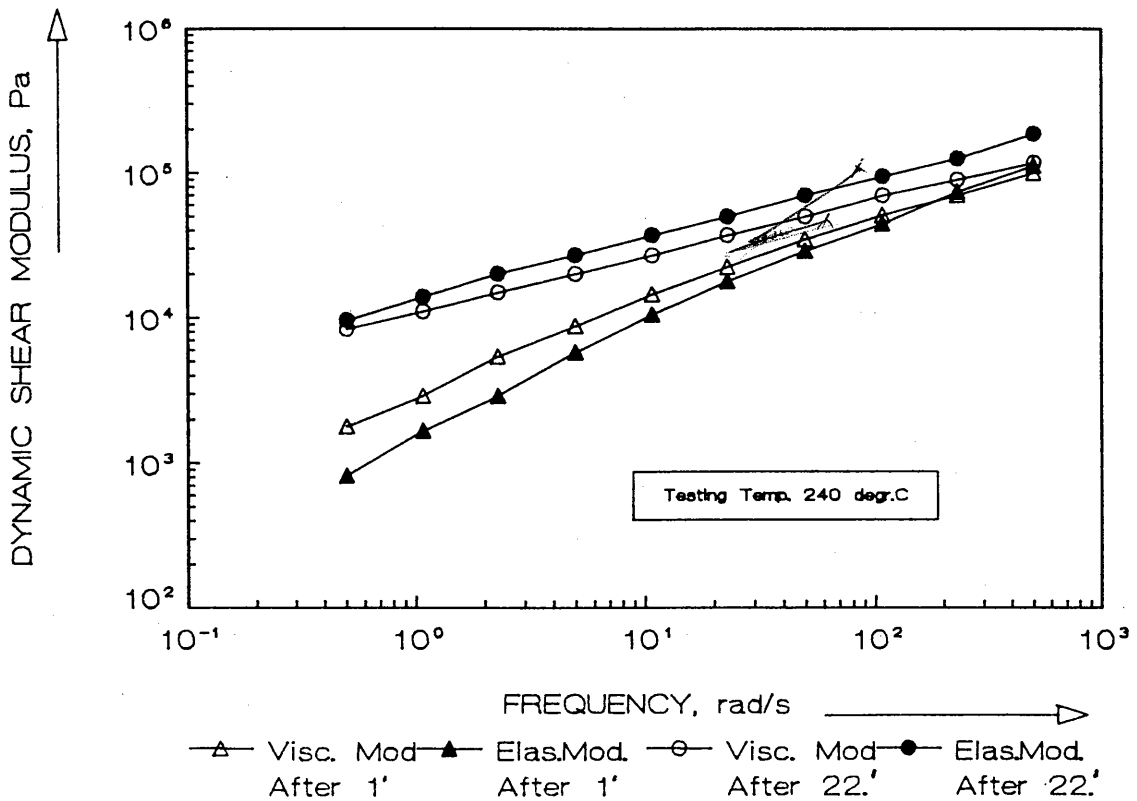
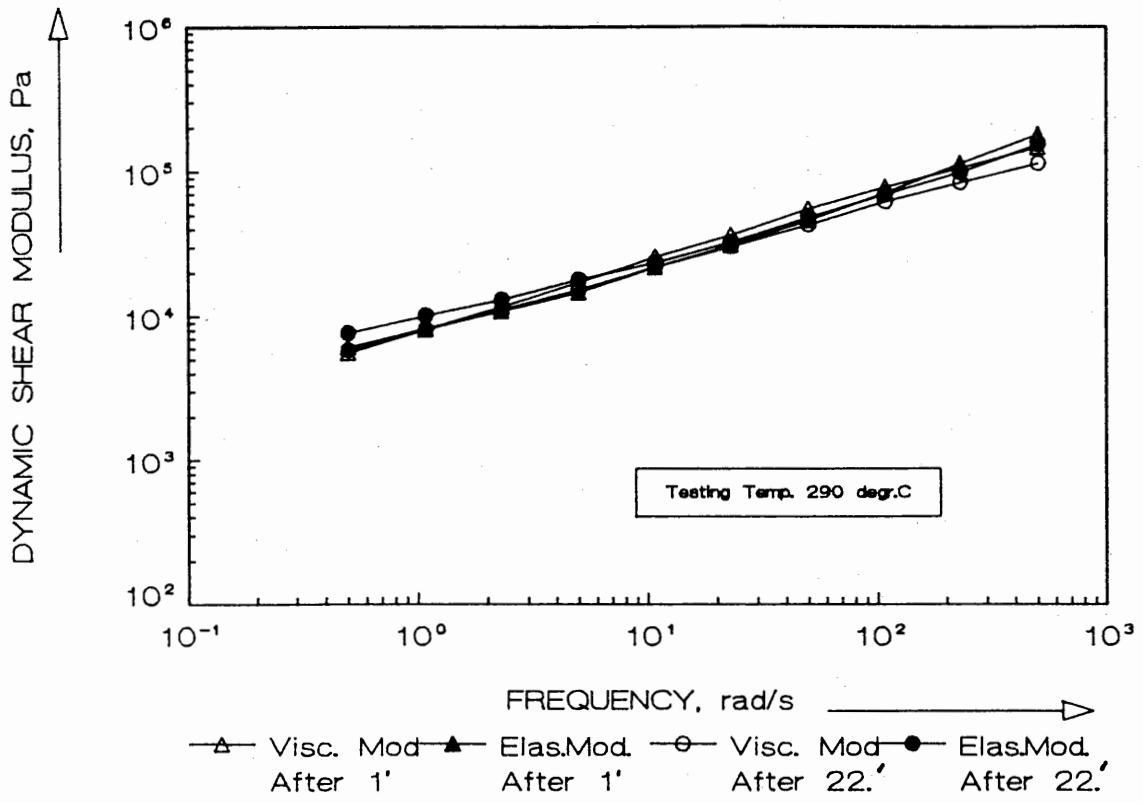


Fig. 4 DYNAMIC VISCOSITY
NYLON / PPO



TMA Normalization: m0018
Sample Height: 2.752 mm
Fri Nov 18 14:39:26 1988
CARILON EP/GLASS POWDER
(Normalized)

PERKIN ELMER
7 Series Thermal Analysis System

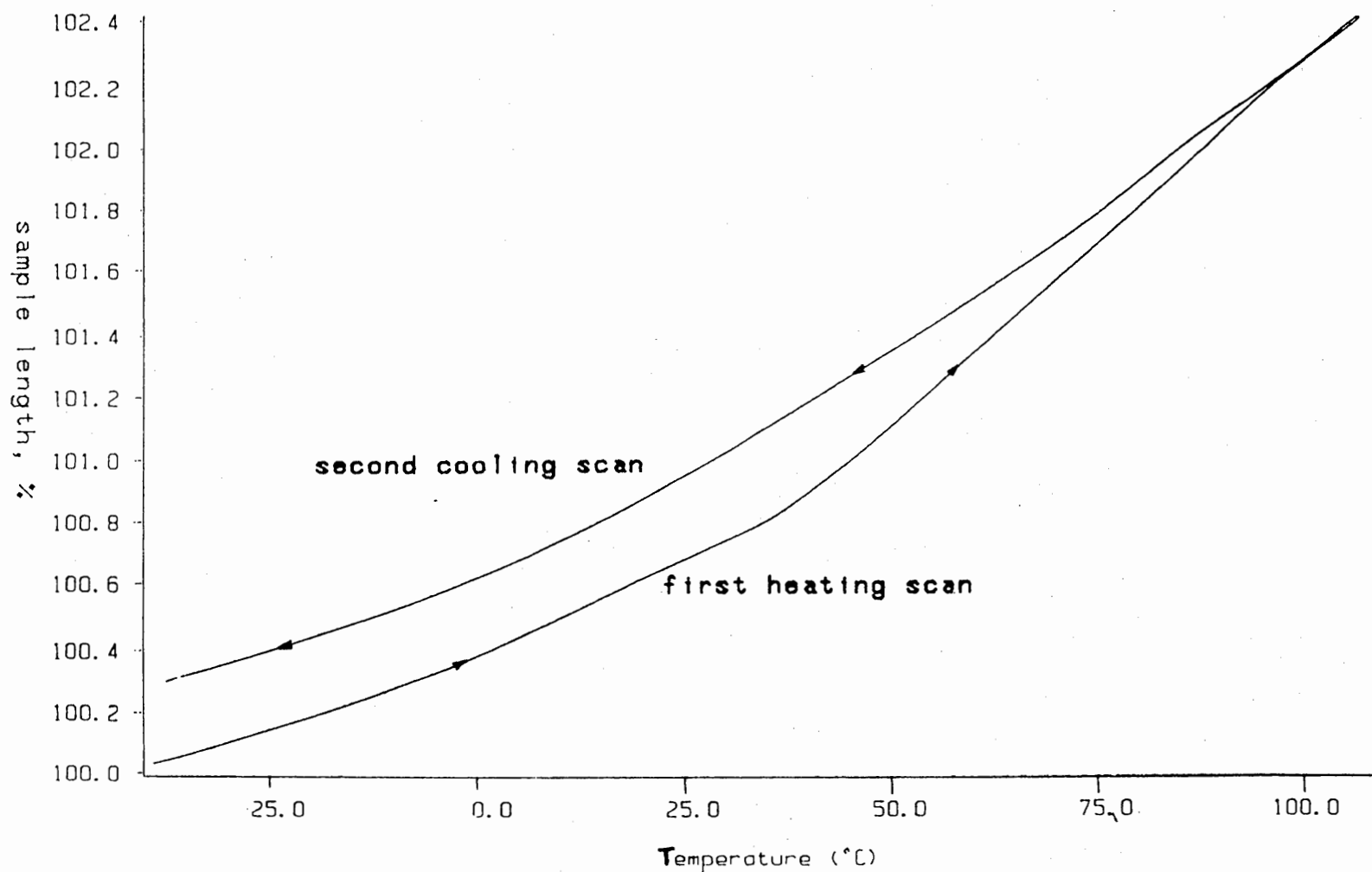


FIGURE 5. LENGTH CHANGE OF THE CARILON EP SYSTEM DURING HEATING FROM -40 C UP TO 100 C AND COOLING AGAIN TO -40 C

IMA Normalization: m0019
Sample Height: 2.497 mm
Tue Nov 22 15:07:09 1988

PERKIN-ELMER
7 Series Thermal Analysis System

SHEET MOULDING COMPOUND (Citroen BX)
(Normalized)

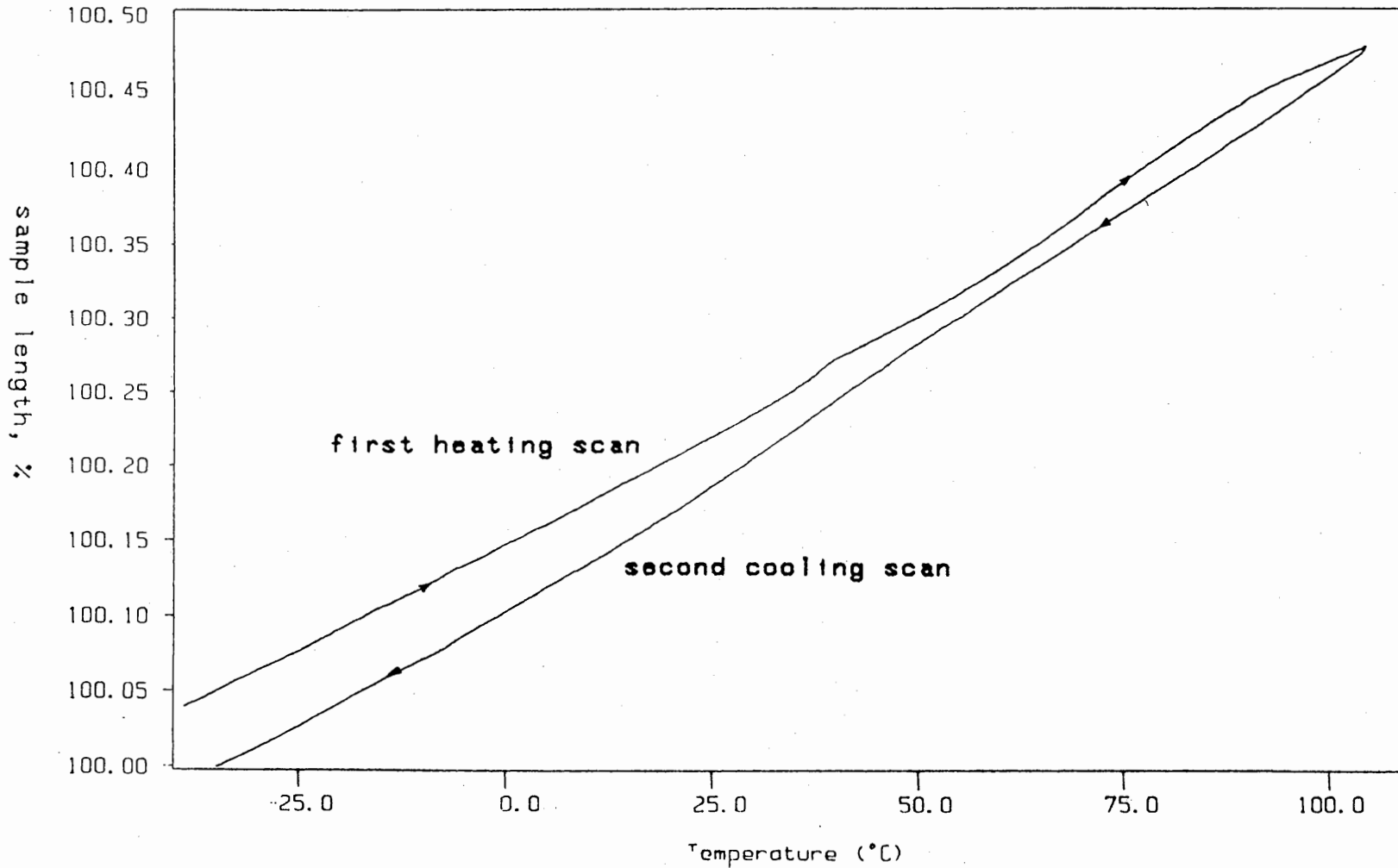


FIGURE 6. THE LENGTH CHANGE OF THE CITROEN SHEET MOULDING SAMPLE (Z-DIRECTION) DURING HEATING FROM -40 C UP TO 100 C AND COOLING AGAIN TO -40 C

TMA Normalization: m0018
Sample Height: 2.752 mm
Fri Nov 18 14:39:26 1988
CARILON EP
(Normalized)

PERKIN-ELMER
7 Series Thermal Analysis System

TMA Normalization: m0019
Sample Height: 2.747 mm
Mon Nov 21 13:10:59 1988
NORYL GTX 910
(Normalized)

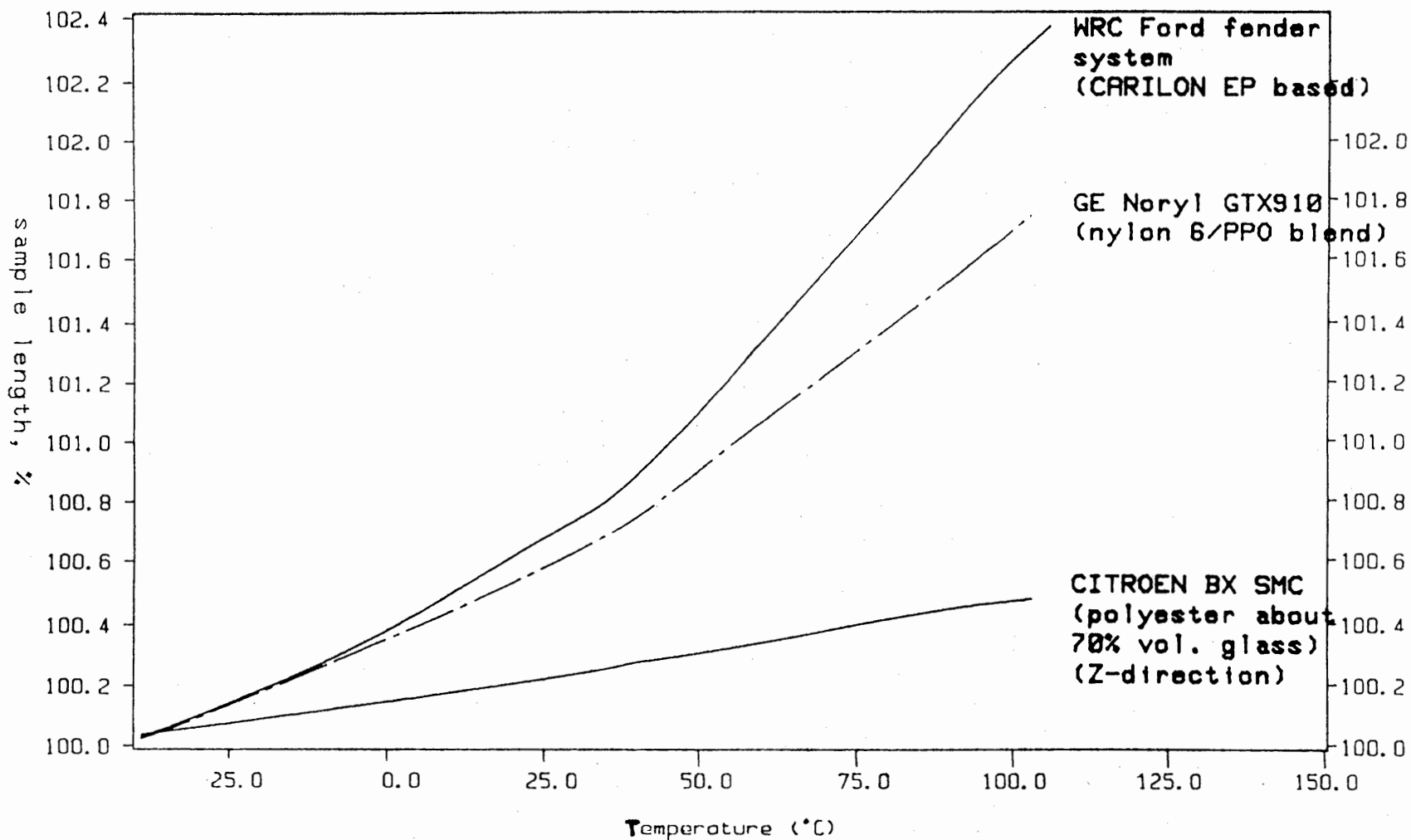


FIGURE 7. CHANGE IN LENGTH OF THE THREE SYSTEMS INVESTIGATED DURING THE FIRST HEATING SCAN

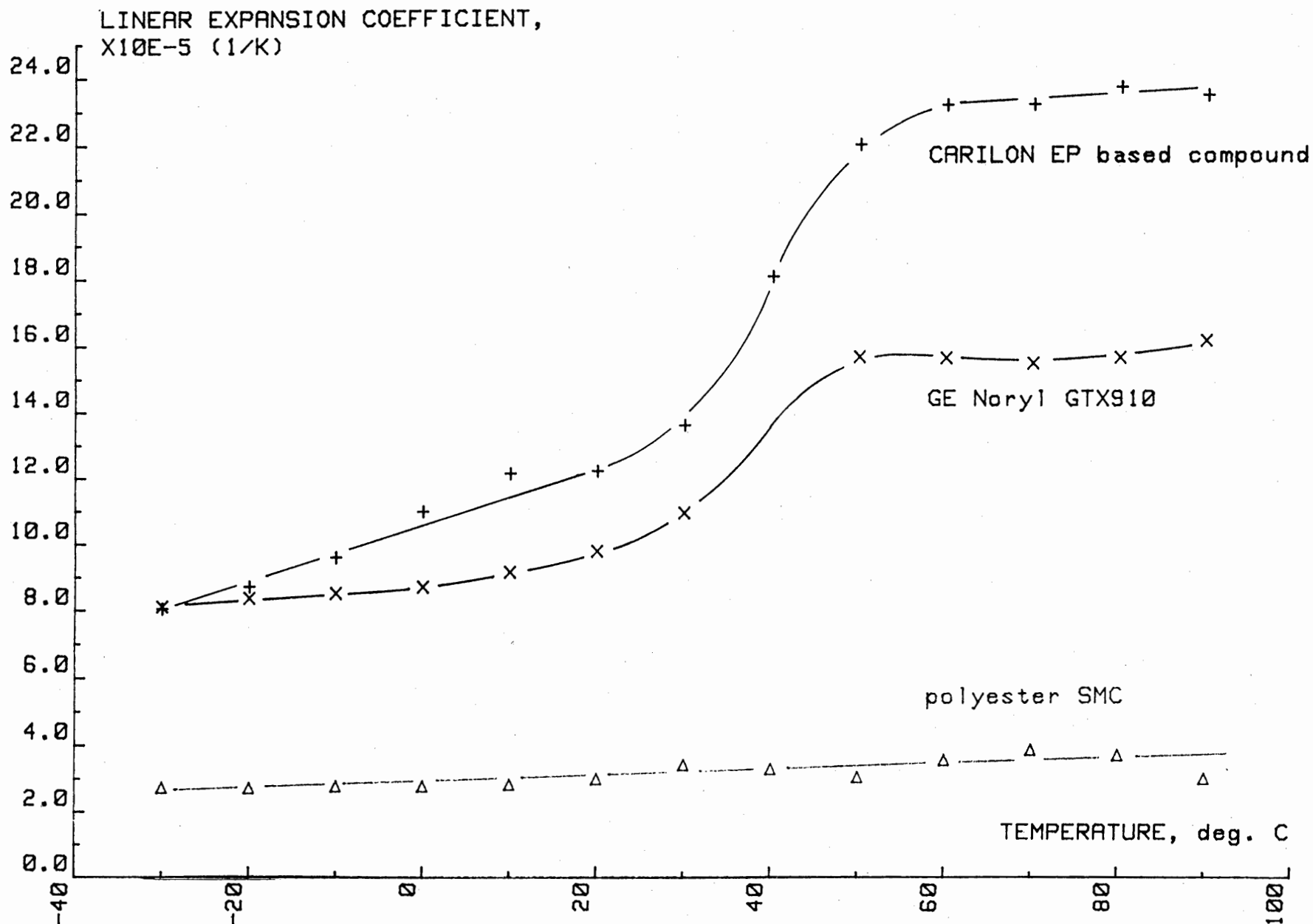


FIGURE 8. LINEAR EXPANSION COEFFICIENT/TEMPERATURE RELATION DURING THE FIRST HEATING SCAN

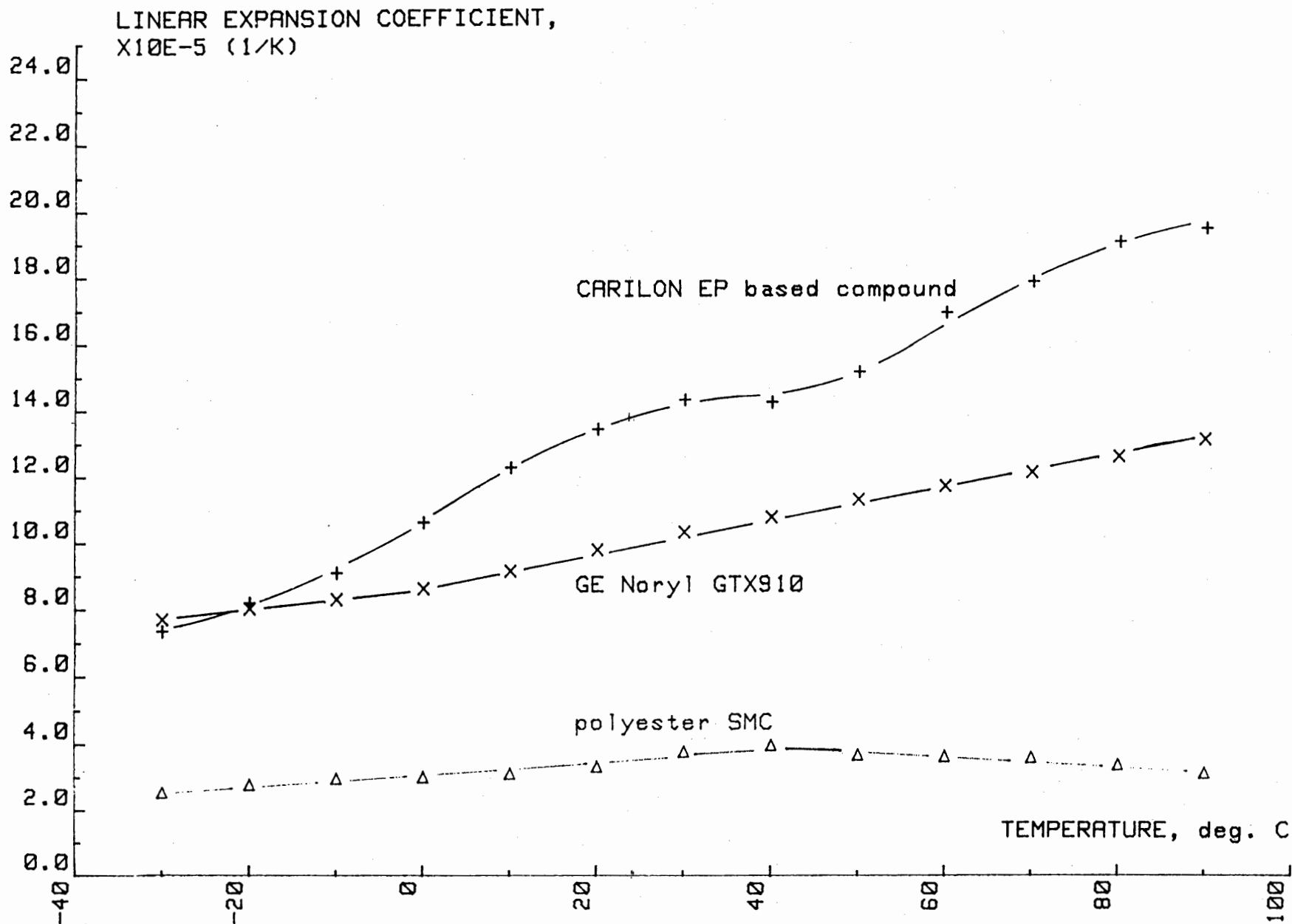


FIGURE 9. LIN. EXPANSION COEFF./TEMPERATURE RELATION DURING THE SECOND HEATING SCAN

TMA Normalization: m0019
Sample Height: 2.497 mm
Tue Nov 22 15:07:09 1988

PERKIN-ELMER

7 Series Thermal Analysis System

TMA Normalization: m0024
Sample Height: 9.586 mm
Tue Dec 06 14:07:47 1988

SHEET MOULDING COMPOUND (Citroen BX)
(Normalized)

SHEET MOULDING COMPOUND Citroen BX (X)
(Normalized)

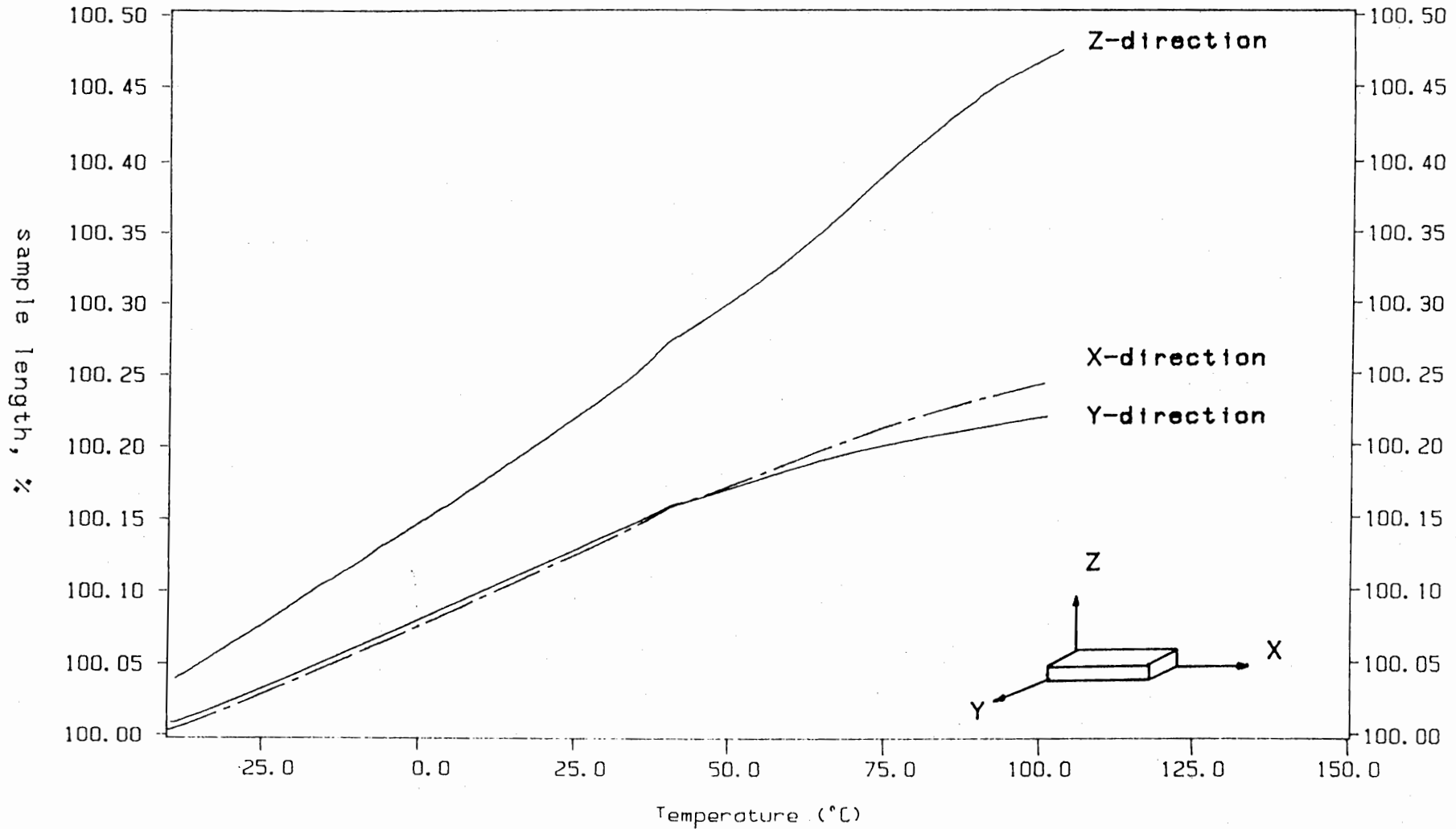
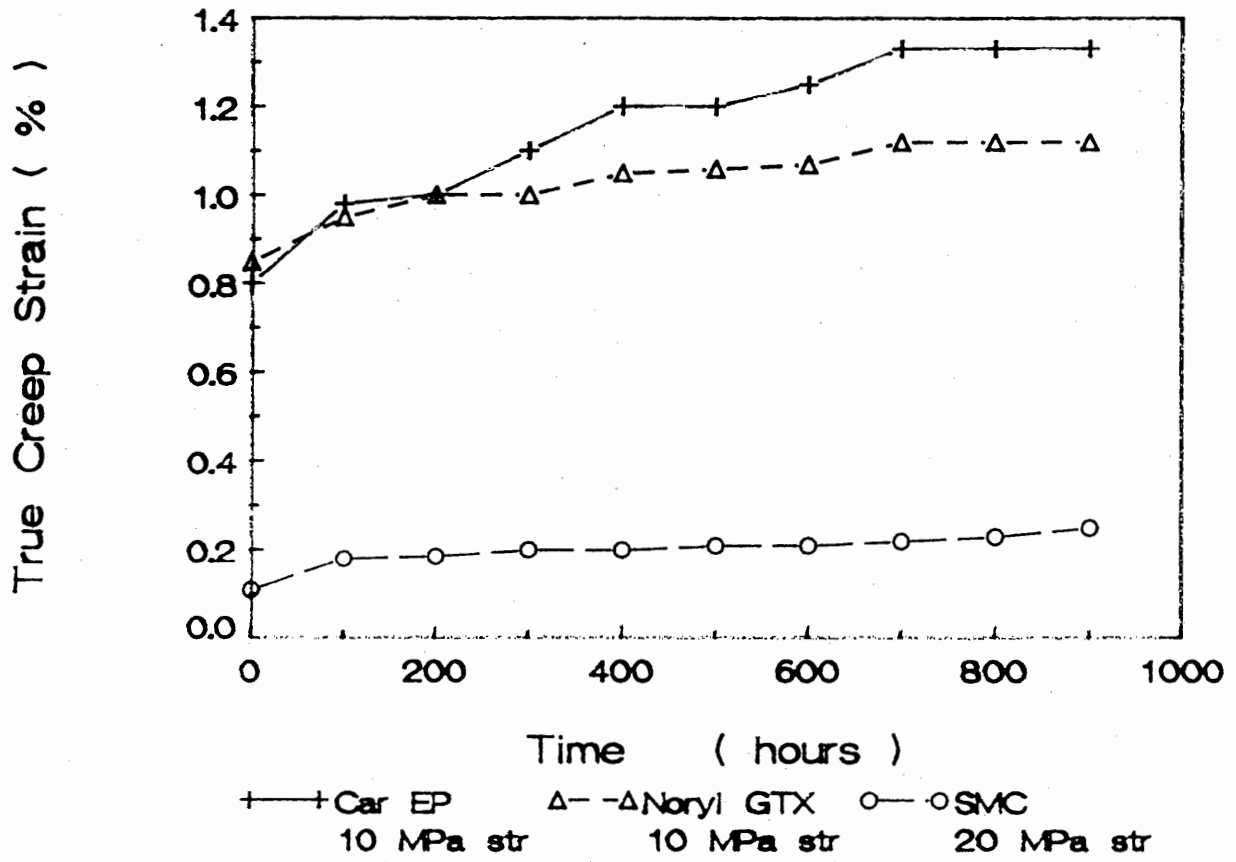


FIGURE 10. CHANGE IN LENGTH OF THE CITROEN BX SMC DURING THE FIRST HEATING SCAN AS A FUNCTION OF THE SAMPLE DIRECTION

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Fig 11. TRUE CREEP STRAIN (%) AT 23 C
At different stress loadings



LIST OF ABBREVIATIONS

CLTE	Coefficient of Linear Thermal Expansion
Polyurethane	RIM PU Reaction Injection Moulding.
EPDM/PP	Ethylene Propylene Diene monomer/Polypropylene
PC	Polycarbonate
PBT	Polybutylene terephthalate
TPE	Thermoplastic polyester
PPO	Polyphenylene oxide
SMC	Sheet moulding compound (polyester)
BMC	Bulk moulding compound (polyester)
ZMC	Dough moulding compound (polyester)
WRC	West Hollow Research Centre
LVN	Limiting Viscosity Number
MPa	Mega Pascal
Tg	Glass transition temperature
HDT	Heat distortion temperature

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