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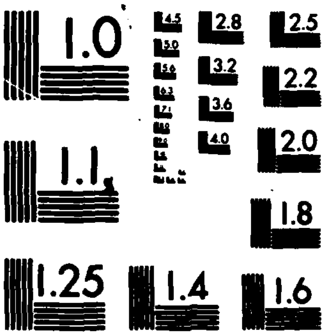

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PROPERTIES OF MULTIPHASE POLYURETHANE SYSTEMS

Final Report for Period July 1, 1982-June 30, 1983

Naval Air Systems Command Code 5304 C2
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July, 1983

(Approved for public release; distribution unlimited)

A. Modified Polyurethanes - Polyether Polyurethaneureas (PEUU's)

1. Introduction

The incorporation of urea linkages in the polyurethane hard segment has a profound effect on the phase separation and domain structure of polyether polyurethaneureas (PEUU's). This is due to the high polarity difference between the hard and soft segments and the likely development of a three dimensional hydrogen bonding network. A new series of PEUU's was synthesized from 4,4'-diphenyl methane diisocyanate, ethylene diamine and poly(tetramethylene oxide)(PTMO). The molar ratios of the reactants and the soft segment (PTMO) molecular weight were varied to produce samples of varying hard segment content and block length. Previously (1981-82) we reported results of thermal, dynamic mechanical and tensile experiments on these materials. In the past year we have continued our investigation of these materials using IR spectroscopy.

2. Results and Discussion

IR survey spectra (Figure 1) indicated that these materials were especially suited for study using IR spectroscopy. Hydrogen bonding shifts the peak position of the participating groups and in several cases allows the distinction of bonded and non-bonded peaks. In particular, the existence of separate carbonyl peaks for the urethane groups ($CO_{B,UT}$(BONDED), $CO_{F,UT}$(FREE)) and the urea groups ($CO_{B,UA}$, $CO_{F,UA}$) provides insight into the domain morphology of these materials. In PEUU's the urethane carbonyls are located only at the junction between the hard and soft segment and thus reflect the morphology of the interfacial region while the urea carbonyls are located within the hard segment and reflect hard segment domain morphology. Increasing hard segment content was found to increase the fraction of hydrogen bonded urethane carbonyls and was attributed to better hard segment domain ordering and a lower percentage of hard segments dissolved in the soft polyether phase. These conclusions were consistent with thermal and mechanical data reported in our 1981-82 final report.

The influence of sample composition on the infrared peak position was investigated for the NH_B (bonded) and $CO_{B,UA}$ groups. The existence of 3-dimensional hydrogen bonding (two C=O groups bonded to one N-H group) should shift the $CO_{B,UA}$ peak to lower frequency and the NH_B peak to higher frequency compared to when they form a conventional hydrogen bond (one C=O group bonded to one N-H group). The results (Figure 2) showed that the PEUU-46 and PEUU-36 samples have mostly three dimensional interurea hydrogen bonding, however, a decrease in hard segment content or, equivalently, an increase in soft segment content results in some soft segments dispersed in the hard domains. This interferes with the ordering of the hard segments and also allows the polyether oxygens to compete with urea carbonyls for the urea NH groups. Both factors result in a mixed state having three dimensional as well as conventional interurea bonds in samples PEUU-25-1000 and PEUU-25-2000. Thus one observes a large shift in average urea carbonyl peak position to lower frequency upon going from samples containing 25 wt% hard segment to 36 wt%.

An analysis of IR absorption as a function of strain was performed to gain information on changes in domain morphology during deformation. The concentration of $CO_{B,UA}$ groups was found to decrease as the sample was elongated for all samples except PEUU-25-1000 and the decrease was at a higher rate for samples of higher hard segment content. The interconnected hard domains in these samples appear to be subjected to the external stress immediately upon sample deformation. This stress disrupts the ordering in the hard domains possibly leading to dissociation of some of the interurea hydrogen bonding. This is consistent with the stress hysteresis data (reported last year) which indicated that samples of higher hard segment concentration and longer block length underwent greater plastic deformation during strain cycling. The NH_B absorption contains contributions from NH groups participating in either interurea, interurethane or hard to soft segment hydrogen bonding. Thus while the change in concentration of NH_B groups with strain was similar to that of the urea carbonyl it decreased to a lesser extent. In contrast to the bonded NH and urea carbonyl both the urethane carbonyl absorptions remained relatively constant upon deformation.

Infrared dichroism experiments were performed to measure segmental orientation during deformation. The CH stretching band was used to measure soft segment orientation while the NH and C=O bands were used to monitor hard segment orientation. In order to study the influence of block length on hard segment orientation, it is instructive to compare IR dichroism results on samples containing similar hard segment concentrations. Stress hysteresis data showed that PEUU-46 samples contained highly interlocked hard segment domains. However, both the DSC and Rheovibron results also indicated that PEUU-46-2000 had higher phase purity and degree of order in both the hard domains and the polyether matrix phase. Figures 3A and B shows that $f_{CO_{B},UA}$ and f_{NH_B} of PEUU-46-1000 are less negative at low strains than those of PEUU-46-2000. It appears that the hard segment lamellae in the 2000 MW sample have better domain cohesion. This arises because PEUU-46-2000 has a higher fraction of urea groups and degree of interurea hydrogen bonding. Therefore the domains in the longer segment material are not disrupted into microdomains containing bundles of hard segments oriented preferably along the stretch direction until higher strain levels. The elongation at the minimum values of $f_{CO_{B},UA}$ or f_{NH_B} may serve as a measure of when large scale domain disruption takes place. A higher value of strain at this minimum is observed for PEUU-46-2000 compared to PEUU-46-1000 (140% vs. 100%). Similar trends of orientation behavior for soft and hard segments within domains or at the interface were observed for sample pairs PEUU-36-1000 and 2000 and PEUU-25-1000 and 2000.

3. Significance

The study of polyether polyurethaneureas can aid in our understanding of how in this system of high hard segment/soft segment polarity difference, the urea linkage, hard segment content, block length and morphology affect physical properties.

Infrared spectroscopy proved to a particularly useful tool to analyze PEUU's. Increasing hard segment content was found to increase the fraction of hydrogen bonded urethane carbonyls and shift the peak positions of the $CO_{B,UA}$ and NH_B groups. These results were interpreted as an increase in hard segment domain ordering and phase separation and as an increase in the extent of 3-dimensional hydrogen bonding respectively. Increasing hard segment content also accelerated the dissociation of $CO_{B,UA}$ groups during deformation. This phenomena was attributed to a change from an isolated to an interconnected hard segment domain morphology. Infrared dichroism experiments indicated that the hard segments within domains initially orient transverse to the stretch direction whereas the soft segments orient parallel to the stretch direction.

B. Environmentally Stable Polyurethanes-Polyisobutylene Based Polyurethanes

1. Introduction

Some of the properties restricting applications of conventional polyether or polyester based polyurethanes are the high moisture permeability and low hydrolytic stability resulting from the ester and ether bond linkages in the soft segment. Thus several new families of polyurethanes based on hydrocarbon soft segments have been recently disclosed. Polybutadiene was the first such hydrocarbon soft segment; it however suffers from poor oxidative stability because of the unsaturated double bond and its analysis is confounded by the existence of three isomers (cis, trans, vinyl). To offset these difficulties while retaining the advantages inherent in hydrocarbon soft segments, we have initiated a study of polyurethanes based on a polyisobutylene soft segment. These materials should be more environmentally stable than either polybutadiene or conventional polyether or polyester based polyurethanes.

2. Results and Discussion

The materials studied were prepared in a standard two step bulk polymerization and were kindly provided by Dr. Gary Ver State of the Exxon Chemical Co. The soft segment diol had less than perfect functionality which resulted in a low molecular weight product material. Environmental tests indicated that the synthesized materials did indeed exhibit improved hydrolytic stability and moisture permeability (by 3 orders of magnitude) compared to polyether or polyester polyurethanes and improved oxidative stability (by an order of magnitude) compared to polybutadiene polyurethanes.

In addition to their environmental stability, polyisobutylene polyurethanes are of interest for structure-property studies because of the large polarity difference between the hard and soft segments and the inability of the soft segments to hydrogen bond to the hard segments. These factors lead to a high degree of segmental incompatibility and provide a large driving force for microphase separation. DSC and dynamic mechanical analysis (Figure 4) indicated a higher degree of phase separation in these materials than that reported for polyether or polyester polyurethanes. This high degree of phase separation is reflected in a soft segment glass transition temperature that does not vary with the sample composition. The dynamic mechanical experiments (Figure 4) also showed that the storage modulus above the soft segment T_G increases with increasing hard segment content, a common phenomenon in phase separated polyurethanes.

The storage modulus above the soft segment glass transition for these polyisobutylene materials is about an order of magnitude higher than that reported for typical conventional polyurethanes. The high modulus can be attributed to the short chain length of the polyisobutylene; polyisobutylene chains are roughly half as long as polyether or polyester chains of the same molecular weight. It has been found that decreasing the molecular weight of the soft segment and thereby its length, increases the plateau modulus due to more restrictions to chain motion as the soft segment becomes shorter. Finally, improved phase separation and domain cohesion because of the larger polarity difference between polyisobutylene and urethane segments compared to that between polyether or polyester and urethane segments may also be a factor leading to higher modulus in polyisobutylene polyurethanes.

The tensile strengths and elongations at break of the polyisobutylene materials were found to be lower than those reported for conventional polyurethanes. This was probably a result of the overall low molecular weight of the materials. Stress-strain experiments also provided evidence for a morphological transition from interconnected hard segment domains (yielding behavior) to isolated hard segment domains (rubbery behavior) with decreasing hard segment content.

Small angle x-ray scattering (SAXS) experiments were performed to analyze the microstructure of the materials. The results supported the findings of the mechanical and thermal experiments. The SAXS data indicated that the materials were highly phase separated. Increasing hard segment content was found to increase domain size. The effect of the synthesis conditions on two samples was also noted. Two of the samples exhibited macroscopic heterogeneities during the reaction and although their compositions were quite similar, one of the samples possessed far superior tensile properties. The SAXS data (Figure 5) were also quite different for the samples and indicated that the sample (MB 2.8/1.8/1) exhibiting better properties had a higher degree of phase separation (area under SAXS curve) and a more well defined domain structure (development of peak in the curve)

3. Significance

Polyisobutylene polyurethanes have many potential applications because of their improved environmental stability compared to polyether, polyester or polybutadiene polyurethanes. They are also of interest for structure-property studies because of their high degree of segmental incompatibility which leads to a high degree of microphase separation.

Experimental results showed trends similar to those observed in other polyurethane systems. Hard segment content was the factor which influenced properties most significantly; increasing hard segment content resulted in higher modulus, lower elongation at break and larger domains. The polyisobutylene polyurethanes were found as expected to have better environmental properties and a higher degree of phase separation compared to conventional polyurethanes. Problems relating to the synthesis of both the soft segment diol and the overall polyurethane resulted in lower molecular weight which decreased tensile properties.

C. Polyurethane Ionomers

1, Introduction

The introduction of ionic groups into the hard segment of a polyurethane elastomer can markedly affect its properties. In a previous study of polytetramethylene oxide (PTMO) based polyurethanes ionomers we found that increasing ionic content improved phase separation and also affected domain cohesion. To better study these effects we have synthesized three series of polyurethane ionomers based on polyethylene oxide (PEO), polypropylene oxide (PPO), and polybutadiene (PBD) soft segments and the same hard segment - diphenylmethane-diisocyanate/N-methyldiethanolamine (MDI/MDEA).

Each polymer was synthesized using a two step reaction technique. The number average molecular weight for each polymer is K 40,000 with a polydispersity K 2.3. The polymer was then sulfonated to varying extents at the tertiary amine of the chain extender using propane sultone. Two different molar ratios of hard segment to chain extender to soft segment each with three levels of ionization were prepared based on the four different soft segment materials.

2. Results and Discussion

DSC experiments were performed to examine the effects of ion incorporation on thermal behavior. Table I lists the glass transition termperature, transition zone width and the WC_p at the glass transition zone for the PEO, PPO and PBD based material. The code used in Table I describes for example HTPBD-PU-30-6.4 as a sample containing hydroxy terminated polybutadiene, 30 wt% MDI and sulfonated to the extent of 6.4%. Table I shows that the incorporation of ionic groups into the polybutadiene polyurethane causes no change in glass transition temperature and transition zone width of the polybutadiene domains. This suggests that the ionic groups reside primarily in the hard segment domains and do not affect the degree of phase separation. Although an increase in phase separation might be expected, previous experiments indicated that the control polymer (no ionic content) was already very well phase separated.

In the polypropylene oxide and polyethyelene oxide polyurethane systems, the soft segment ether groups have a strong tendency to form hydrogen bonds with the urethane N-H groups. Therefore, phase mixing in these two systems of polyether polyurethane ionomers is expected. As shown in Table I a decrease in the glass transition temperature and width of the glass transition zone was caused by the incorporation of ionic groups. This is due to increased phase separation. Pendant ionic groups on the backbones of hard segments produce a higher affinity among hard segments through ionic interaction. Therefore, a well phase separated morphology with strong domain cohesion is expected.

To complement the thermal analysis, dynamic mechanical analysis of these materials was carried out. Figures 6 and 7 show the results for HTPBD and PEG series of polyurethane zwitterionomers. In accord with the DSC data, the incorporation of ionic groups caused no change of the soft segment glass transition temperature (E" peak) of polybutadiene based polyurethanes suggesting no effect on the phase separation. However, the rubbery modulus above the soft segment T_g (-50°C) increases with increasing degree of ammonium sulfonation. For example, the sample HTPBD-PU-37-8.4, which possesses 8.4% by weight of sulfonate groups, exhibited an extended plateau region up to 200°C (the parent material (HTPBD-PU-37-0) becomes soft at 50°C).

In contrast with the polybutadiene based zwitterionomers, the polyethylene oxide based polyurethane zwitterionomers display a lower soft segment glass transition temperature (Figure 5) than the parent material (PEG-PU-37-0) indicating an improvement in phase separation. As shown by the E' curves, the modulus of PEG-PU-37-0) drops rapidly at the glass transition zone and becomes too low to be measured in the rubbery state by our apparatus. However, as ionic groups are incorporated, a rubbery plateau develops suggesting formation of a two phase morphology. It was also found that the incorporation of ionic groups caused a broadening of the b peak which is associated with soft segment relaxation in the glass transition region. This may be ascribed to a strong interaction between the PEO soft segment phase and the ionic hard segment phase.

Stress-strain data are presented in Table II for the PBD, PTMO and PPO based materials. As would be expected based on the dynamic mechanical data, Young's modulus and the stress at break increased with increasing hard segment content and increasing ionic content. Again this is the result of improved phase separation and domain cohesion. Table I also shows that the increases in strength were obtained by sacrificing the materials extensibility.

Infrared dichroism experiments indicated that increasing ionic content led to increasing orientation of both the soft and hard segments in the stretch direction. This is primarily a result of improved phase separation. Also, segments orienting into the stretch direction will increase the tensile modulus of the material while likely decreasing its elongation. This in agreement with the observed stress-strain behavior.

3. Significance

This study of polyurethane ionomers has led to a better understanding of how the incorporation of ionic groups improves the physical properties of polyurethane ionomers. In materials that already possess a high degree of phase separation the ionic groups reside primarily in the hard segment domain and improve its cohesion. In materials with a lower degree of phase separation the insertion of ionic groups helps increase the driving force for and improves phase separation. In either case better physical properties such as dynamic and tensile modulus result from ionization. Increasing ionic content also leads to a greater orientation into the stretch direction of both the hard and soft segments.

D. Publications Resulting from this Research January 1982-present

Koshiba, M., K.K.S. Hwang, S.K. Foley, D.J. Yarusso, and S.L. Cooper, "Properties of U.V. Curable Polyurethane Acrylates", J. Mater. Sci., 14, 1447 (1982).

Wang, Carl B., and S.L. Cooper, "Segmental Orientation in Multicomponent Polymer Systems", ACS Advances in Chemistry Series, Ed. C.D. Han, ACS 1982, in press.

Yang, C.Z., K.K.S. Hwang, and S.L. Cooper, "Morphology and Properties of Polybutadiene and Polyether-Polyurethane Zwitterionomers, Die Makromolekulare Chemie, 184, 651 (1983).

Wang, C.B. and S.L. Cooper, "Morphology and Properties of Segmented Polyether Polyurethaneureas", Macromolecules, 16, 775 (1983).

Wang, Carl B. and S.L. Cooper, "Morphology and Properties of Poly(vinylchloride)-Poly(butadiene-co-acrylonitrile) Blends", J. Polymer Sci., Polymer Phys. Ed., 21, 11 (1983).

Miller, J.A., K.K.S. Hwang, and S.L. Cooper, "Properties of Polyether-Polyurethane Anionomers", J. Macromol. Sci.-Phys., B22, 321 (1983).

Speckhard, T.A., G. ver Strate and S.L. Cooper, "Properties of Polyisobutylene-Polyurethane Block Copolymers: I. Macroglycols from Ozonolysis of Isobutylene-Isoprene Copolymer", Polymer Engr. and Sci., 23, 337 (1983).

Hwang, Kirk K.S., D.J. Hemker and S.L. Cooper, "Phase Diagrams and Morphology of a Urethane Model Hard Segment and Polyether Macroglycols," Macromolecules, submitted for publication.

TABLE I

DSC DATA

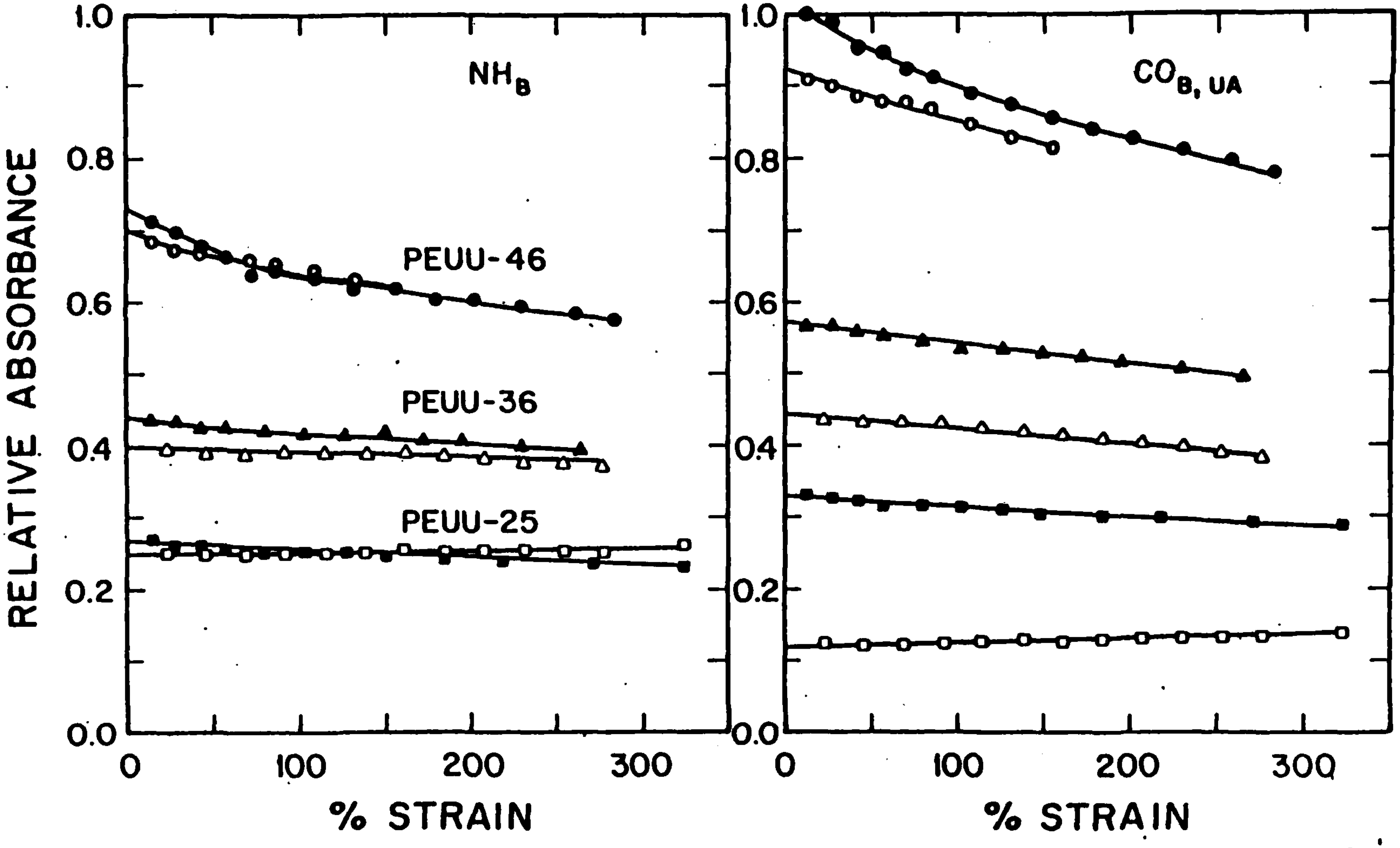
	T_g (°K) MIDPOINT ($\frac{1}{2}\Delta C_p$)	TRANSITION ZONE	ΔC_p
HTPBD-1970	208	206-211 (5)	12.13E-02
HTPBD-PU-30-0	218	212-222 (10)	5.89E-02
HTPBD-PU-30-3.2	218	213-223 (10)	5.77E-02
HTPBD-PU-30-6.4	218	213-223 (10)	5.49E-02
HTPBD-PU-37-0	217	212-222 (10)	4.73E-02
HTPBD-PU-37-4.2	217	212-222 (10)	4.35E-02
HTPBD-PU-37-8.4	218	213-223 (10)	3.86E-02
PPG-2000	206	204-208 (4)	15.82E-02
PPG-PU-30-0	240	230-249 (19)	8.83E-02
PPG-PU-30-3.2	226	220-232 (12)	7.17E-02
PPG-PU-30-6.4	223	219-228 (9)	6.11E-02
PPG-PU-37-0	257	249-270 (21)	6.74E-02
PPG-PU-37-4.2	230	222-237 (15)	4.33E-02
PPG-PU-37-8.4	221	215-227 (12)	3.59E-02
PEG-1500			
PEG-PU-30-0	250	244-255 (11)	12.39E-02
PEG-PU-30-2.9	245	237-252 (15)	11.43E-02
PEG-PU-30-5.8	238	230-245 (15)	8.62E-02
PEG-PU-37-0	269	263-276 (13)	8.52E-02
PEG-PU-37-4.0	256	247-265 (18)	6.22E-02
PEG-PU-37-8.0	243	232-254 (22)	4.66E-02

Table II--Tensile Properties

Sample	Stress at failure (Pa)X10^{-6}	Elongation at failure (%)	Young's Modulus (Pa)X10^{-6}
HTPBD-PU-30-0	5.3	350	17.0
HTPBD-PU-30-3.2	18.0	300	20.0
HTPBD-PU-30-6.4	26.0	250	24.0
HTPBD-PU-37-0	11.0	210	61.0
HTPBD-PU-37-4.2	25.0	180	92.0
HTPBD-PU-37-8.4	26.0	90	150.0
PTMO-PU-30-0	0.46	1940	0.91
PTMO-PU-30-3.2	37.5	720	18.9
PTMO-PU-30-6.4	35.2	480	59.5
PTMO-PU-37-0	7.0	2030	2.7
PTMO-PU-37-4.2	36.9	455	155.0
PTMO-PU-37-8.4	41.4	340	258.0
PPG-PU-30-0	1.0	120	3.3
PPG-PU-30-3.2	9.0	360	7.0
PPG-PU-30-6.4	17.0	440	10.0
PPG-PU-37-0	1.0	300	4.1
PPG-PU-37-4.2	24.0	330	42.0
PPG-PU-37-8.4	33.0	190	84.0

FIGURE CAPTIONS

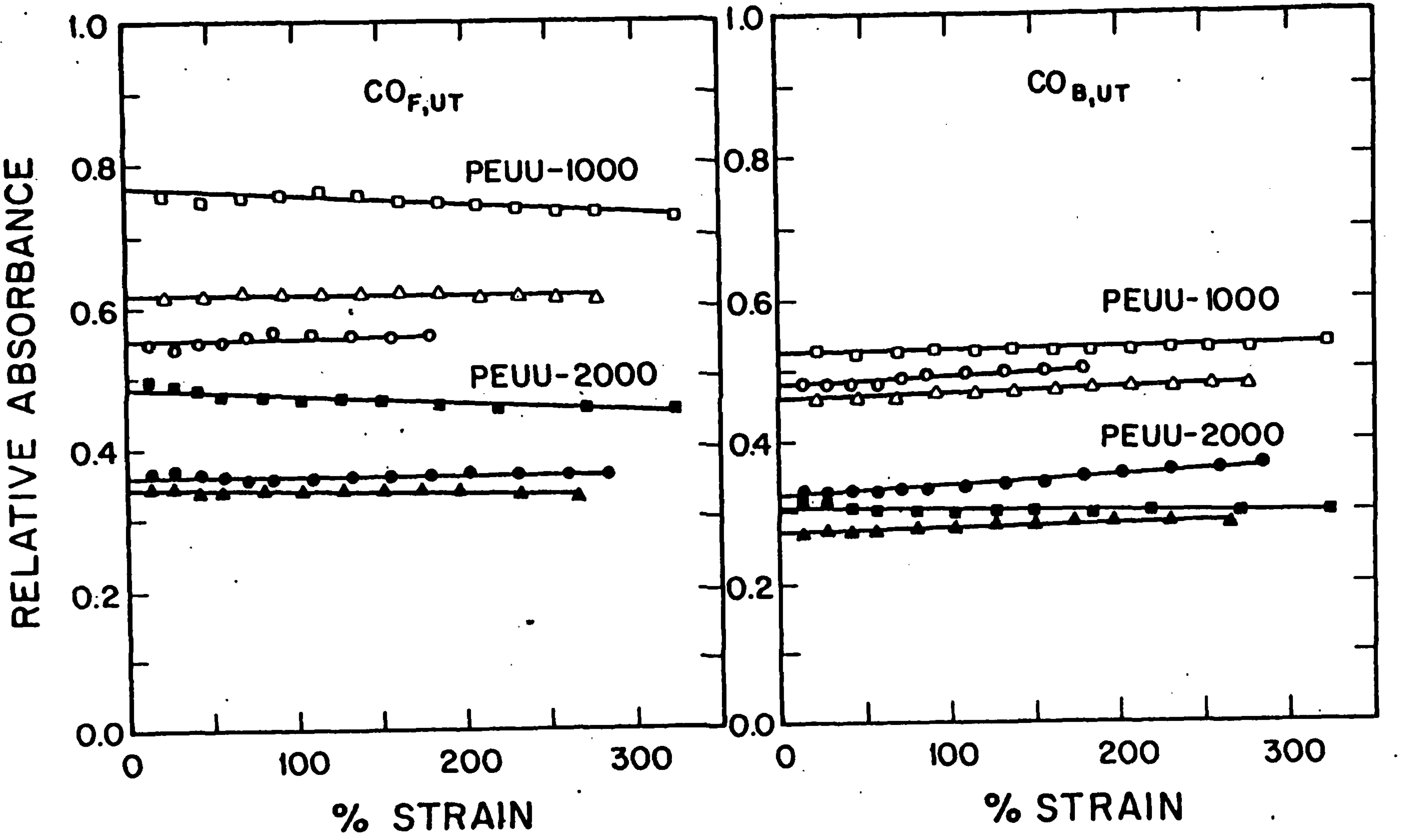
FIGURE 1	IR survey spectra of polyurethaneurea samples PEUU-46-1000 and PEUU-25-1000.
FIGURE 2	Infrared peak positions of the PEUU-1000 and PEUU-2000 series materials.
FIGURE 3	Orientation function vs. elongation curves of (A) PEUU-46-1000 and (B) PEUU-46-2000.
FIGURE 4	Dynamic mechanical data for TP series polyisobutylene materials (E'-storage modulus, tan w-loss factor)
FIGURE 5	SAXS curves for MB series polyisobutylene materials
FIGURE 6	Dynamic mechanical data for polybutadiene based ionomers (E'-storage modulus, E"-loss modulus)
FIGURE 7	Dynamic mechanical data for polyethyleneoxide based ionomers (E'-storage modulus, E"-loss modulus)


RELATIVE ABSORBANCE
1.0
0.8
0.6
0.4
0.2
0.0
0
100
200
300
% STRAIN
NH_B
PEUU-46
PEUU-36
PEUU-25
$CO_{B,\,UA}$

(A)

(B)

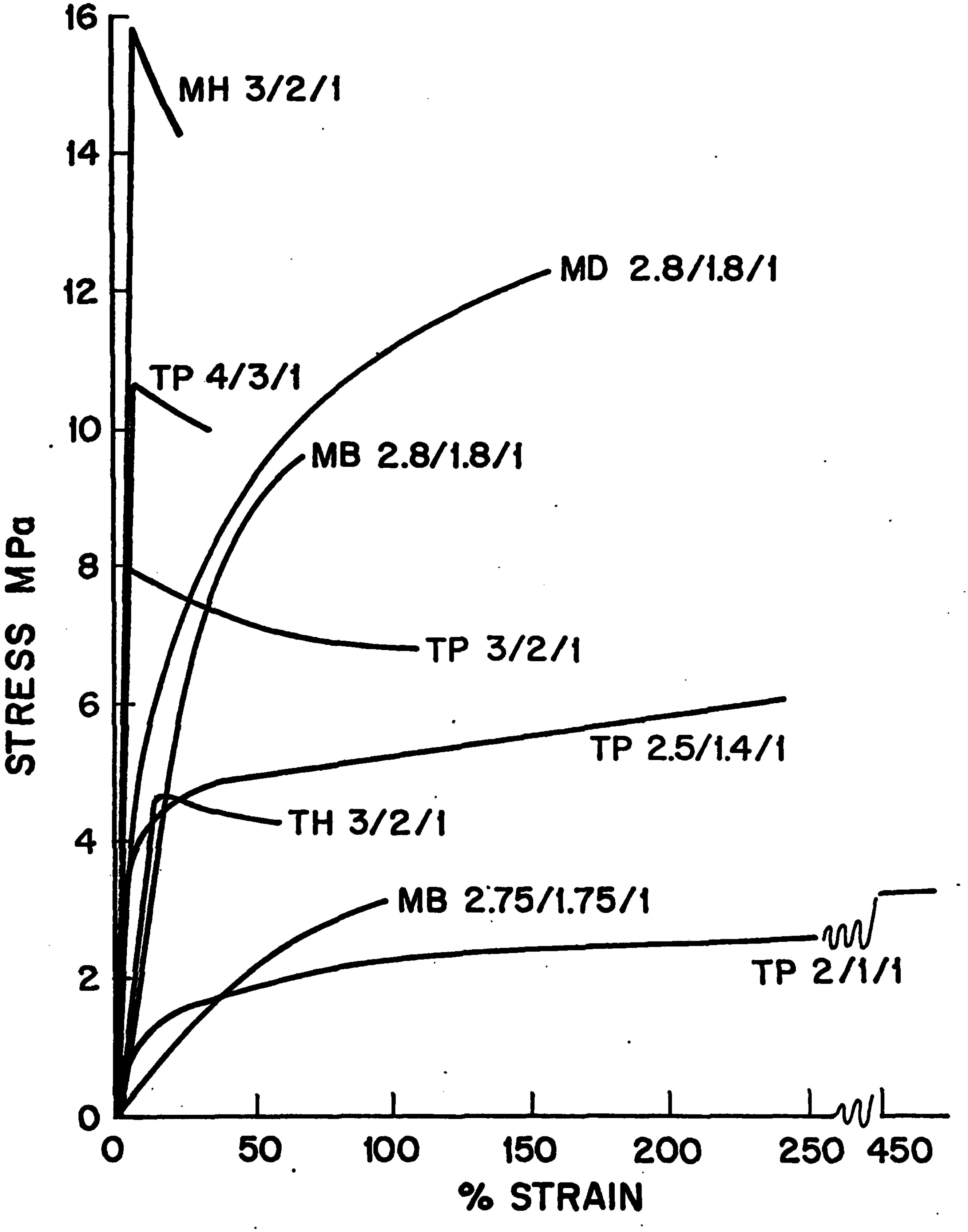
FIG 1


RELATIVE ABSORBANCE
1.0
0.8
0.6
0.4
0.2
0.0
0
100
200
300
% STRAIN
$CO_{F,UT}$
PEUU-1000
PEUU-2000
$CO_{B,UT}$
PEUU-1000
PEUU-2000

(A)

(B)

FIG 2



STRESS MPa
% STRAIN
16
14
12
10
8
6
4
2
0
0
50
100
150
200
250
450
MH 3/2/1
MD 2.8/1.8/1
TP 4/3/1
MB 2.8/1.8/1
TP 3/2/1
TP 2.5/1.4/1
TH 3/2/1
MB 2.75/1.75/1
TP 2/1/1

FIG 3

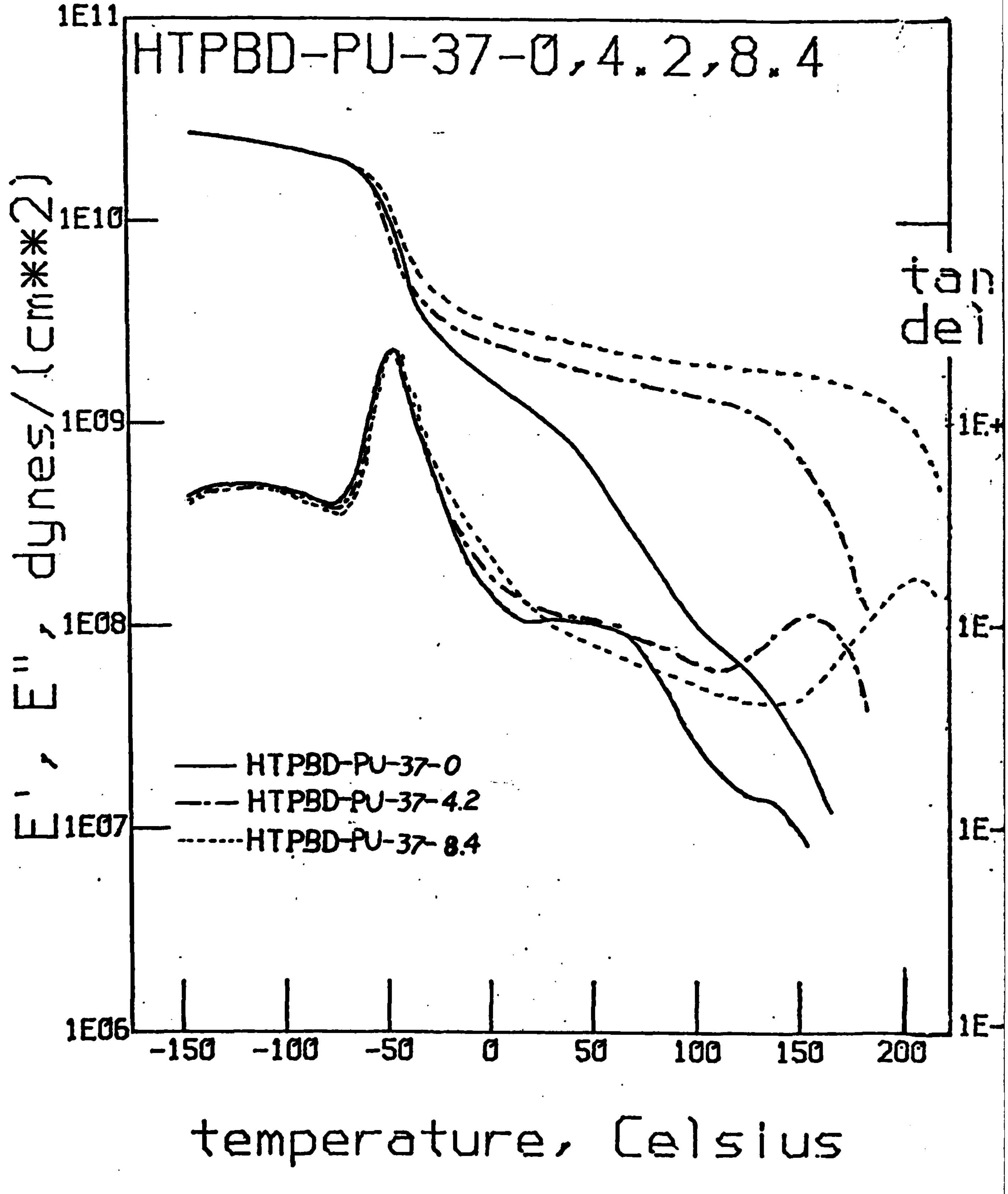

HTPBD-PU-37-0,4.2,8.4
1E11
1E10
1E09
1E08
1E07
1E06
E', E'', dynes/(cm**2)
tan
del
1E+
1E-
1E-
1E-
1E-
HTPBD-PU-37-0
HTPBD-PU-37-4.2
HTPBD-PU-37-8.4
-150
-100
-50
0
50
100
150
200
temperature, Celsius

Fig 4

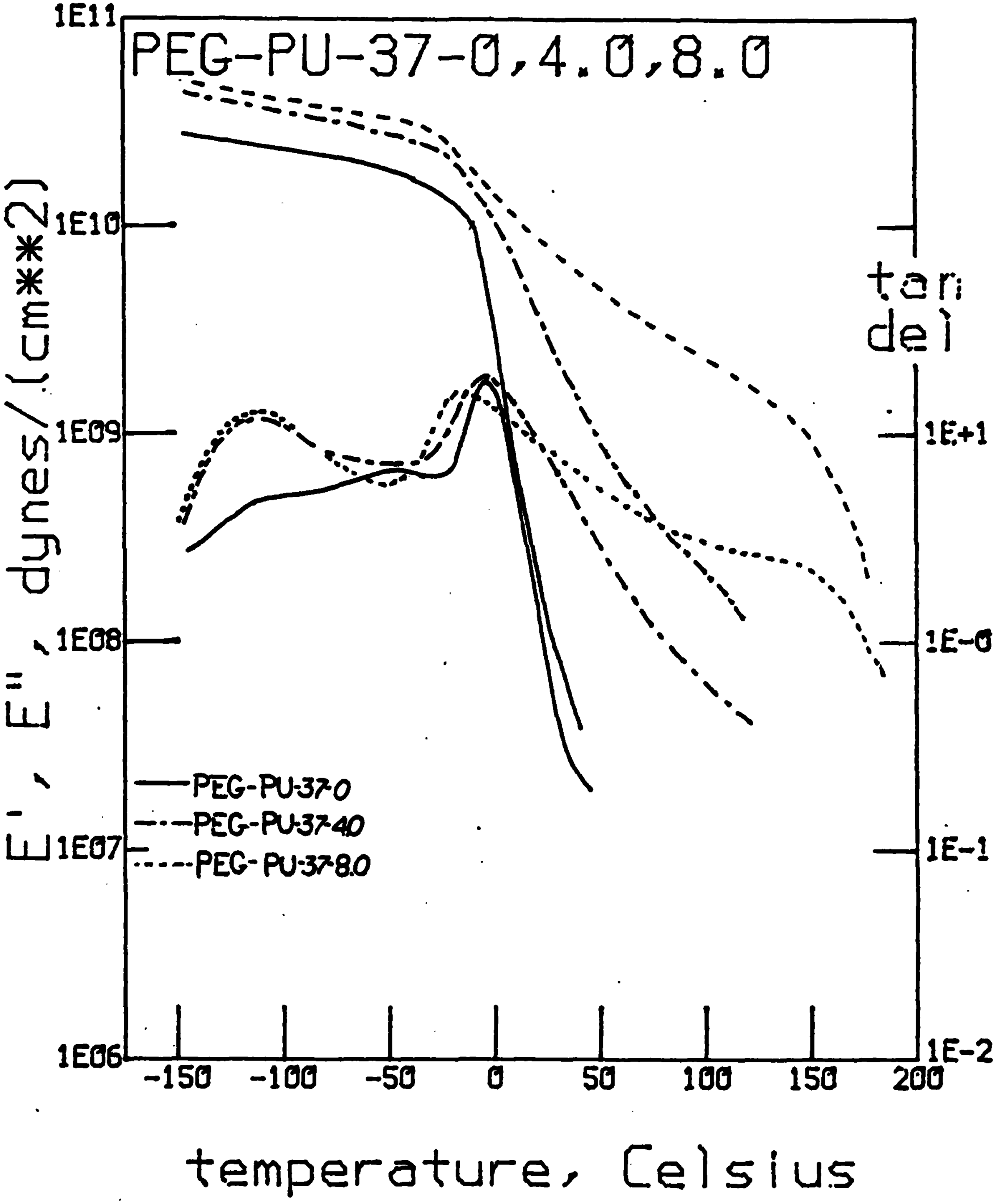

PEG-PU-37-0,4.0,8.0
E', E", dynes/(cm**2)
tan del
temperature, Celsius
1E11
1E10
1E09
1E08
1E07
1E06
1E+1
1E-0
1E-1
1E-2
-150
-100
-50
0
50
100
150
200
PEG-PU-37-0
PEG-PU-37-4.0
PEG-PU-37-8.0

Fig. 5

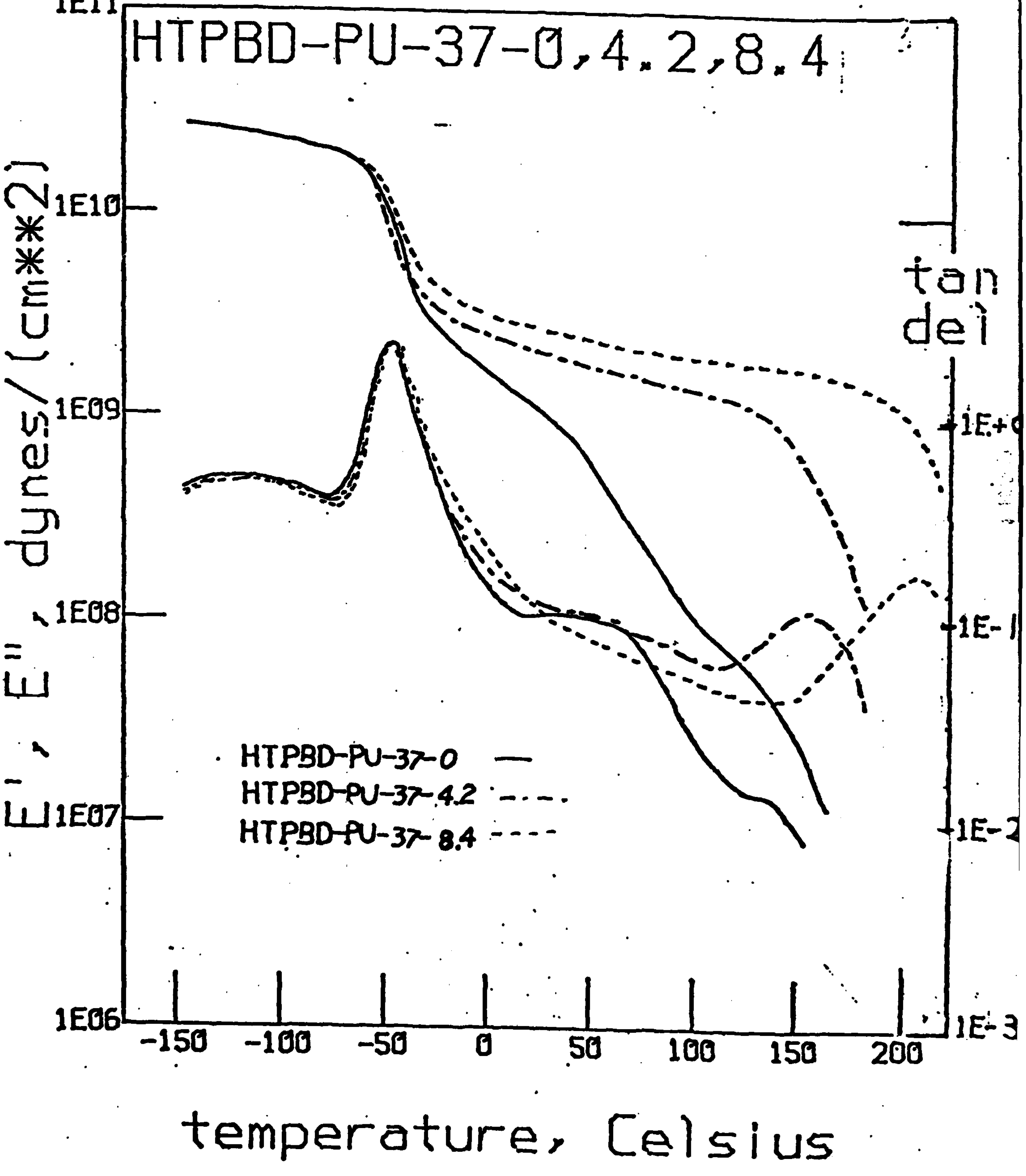
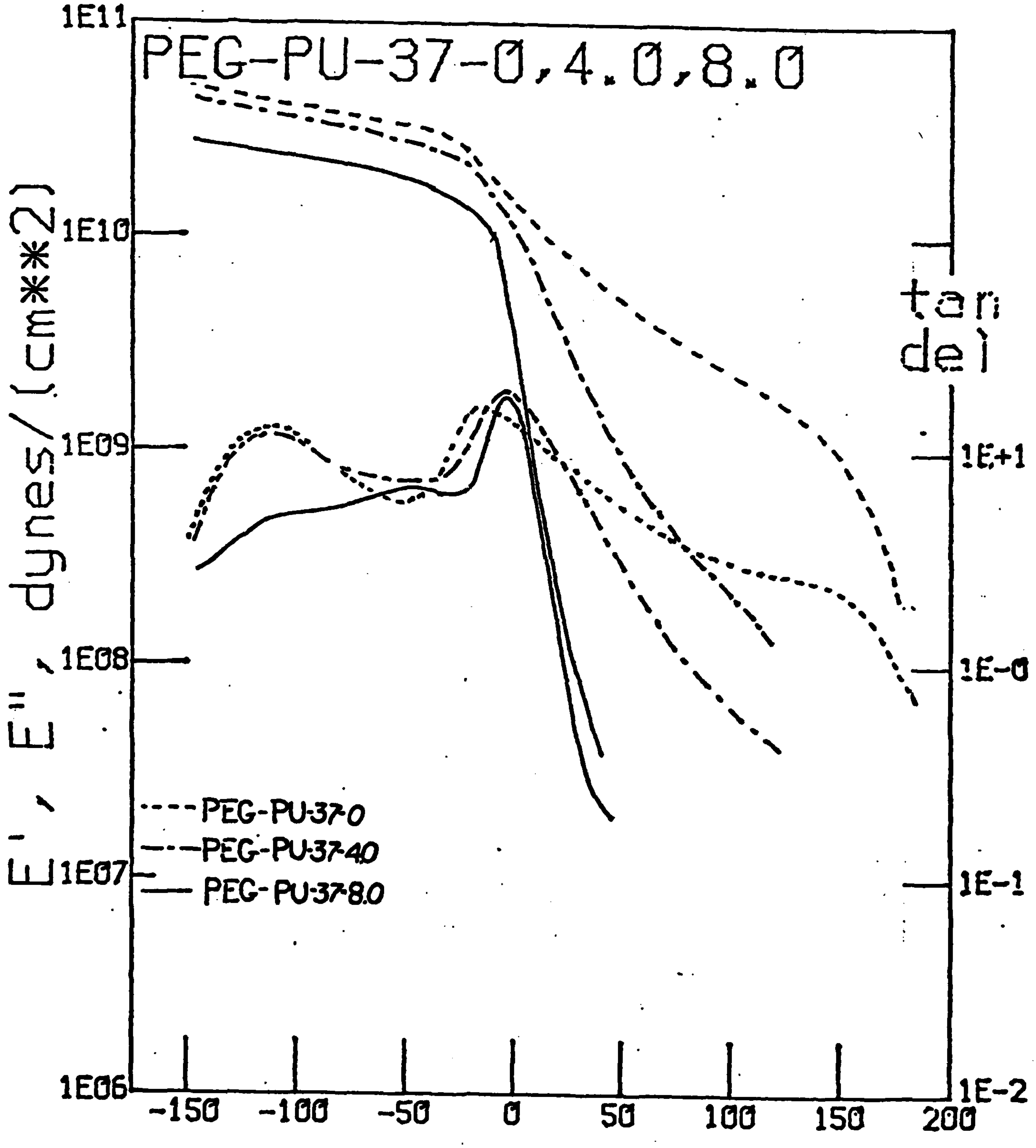

HTPBD-PU-37-0,4.2,8.4
E', E'', dynes/(cm**2)
1E11
1E10
1E09
1E08
1E07
1E06
tan
del
1E+
1E-1
1E-2
1E-3
-150
-100
-50
0
50
100
150
200
temperature, Celsius
HTPBD-PU-37-0 ——
HTPBD-PU-37-4.2 -·-·-
HTPBD-PU-37-8.4 - - - -

FIG 6


PEG-PU-37-0,4.0,8.0
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1E06
E', E", dynes/(cm**2)
tan del
1E+1
1E-0
1E-1
1E-2
-150
-100
-50
0
50
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150
200
temperature, Celsius
PEG-PU-37-0
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FIG 7

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