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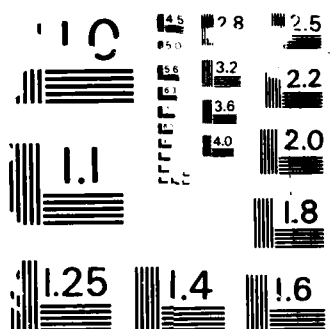
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Electrical D. C. Conductivity of Pitch

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30 October 1987

Prepared for
SPACE DIVISION
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This report was submitted by The Aerospace Corporation, El Segundo, CA 90245, under Contract No. F04701-85-C-0086 with the Space Division, P.O. Box 92960, Worldway Postal Center, Los Angeles, CA 90009. It was reviewed and approved for The Aerospace Corporation by R. W. Fillers, Director, Materials Sciences Laboratory.

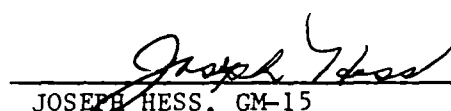
Lt Donald Thoma/CLVT was the project officer for the Mission-Oriented Investigation and Experimentation (MOIE) Program.

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SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

1a REPORT SECURITY CLASSIFICATION Unclassified			1b. RESTRICTIVE MARKINGS		
2a SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited.		
2b DECLASSIFICATION/DOWNGRADING SCHEDULE					
4. PERFORMING ORGANIZATION REPORT NUMBER(S) TR-0086A(2935-06)-1			5. MONITORING ORGANIZATION REPORT NUMBER(S) SD-TR-87-58		
6a. NAME OF PERFORMING ORGANIZATION The Aerospace Corporation Laboratory Operations	6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION Space Division			
6c. ADDRESS (City, State, and ZIP Code) El Segundo, CA 90245		7b. ADDRESS (City, State, and ZIP Code) Los Angeles Air Force Station Los Angeles, CA 90009-2960			
8a. NAME OF FUNDING/SPONSORING ORGANIZATION	8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER F04701-85-C-0086-P00016			
8c. ADDRESS (City, State, and ZIP Code)		10. SOURCE OF FUNDING NUMBERS			
		PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.
11. TITLE (Include Security Classification) Electrical D.C. Conductivity of Pitch					
12. PERSONAL AUTHOR(S) Gold, Phillip I.					
13a. TYPE OF REPORT	13b. TIME COVERED FROM TO	14. DATE OF REPORT (Year, Month, Day) 1987 October 30	15. PAGE COUNT 23		
16. SUPPLEMENTARY NOTATION					
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB-GROUP	Pitch		
			Electrical Conductivity, Activation Energy		
			Mesophase, Rheology		
19. ABSTRACT (Continue on reverse if necessary and identify by block number) Untreated A240 pitch was thermally cycled to 450°C at rates of 5°C/min, 1°C/min, and 5°C/hr. The electric (d.c.) conductivity measured during both heating and cooling portions of the cycle displayed a hysteresis effect resulting from volatilization of lower molecular weight components, polymerization, and mesophase formation. The heating curve obeyed an Arrhenius relation at low temperatures below the onset of significant volatilization, while the cooling curve was observed to follow an Arrhenius relation at all temperatures. Activation energies were similar for heating and cooling.					
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT ☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT ☐ DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION Unclassified		
22a. NAME OF RESPONSIBLE INDIVIDUAL			22b. TELEPHONE (Include Area Code)	22c. OFFICE SYMBOL	

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I. OBJECTIVES

The rheological characteristics of molten pitch are important parameters in the study of the mechanisms of mesophase formation at elevated temperatures and pressures. While it is difficult to measure viscosity under the conditions of temperature and pressure of importance in carbon-carbon impregnation cycles, electric d.c. conductivity is relatively easy to measure under these conditions. It is the objective of this research to investigate the d.c. conductivity of molten pitch as a convenient in situ means of studying pitch rheology.

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II. BACKGROUND

The viscosity of molten pitch is an important design parameter in the development of high-pressure carbon-carbon impregnation cycles. Although pitch may behave as a Newtonian fluid, especially at low temperatures and low rates of shear, the complex physical and chemical processes which occur upon heating and the application of high pressure render it difficult to establish a simple rheological characterization.¹⁻¹⁶

A primary factor influencing liquid pitch properties is the volatilization of lower molecular weight components, followed by polymerization and mesophase formation at elevated temperatures. The mesophase component is non-Newtonian, thixotropic, and viscoelastic and may also exhibit irreversible changes in apparent viscosity as a result of shear working. Moreover, the residual isotropic fraction undergoes changes due to volatilization and polymerization.

A detailed summary of the recent literature of the rheology of pitch can be found in a published report of The Aerospace Corporation¹⁰ and will not be repeated here. Let it be sufficient to note that process design of impregnation cycles requires a significantly more quantitative knowledge of the rheological characteristics of molten pitch than as yet exists.

It was pointed out in a previous report¹⁷ that suitable means are not as yet available at Materials Sciences Laboratory (MSL) to conduct viscometric measurements of this type. On the other hand, it was shown in that report that the electric d.c. conductivity of liquids may be related to fluid viscosity in a manner which makes it a potentially useful parameter with which to study the effect of temperature on rheological characteristics. Moreover, the conductivity property measurement can be performed with considerably more ease than typical viscometric measurements, particularly under the conditions of temperature and pressure of interest here.

The bulk of this report is concerned with the results of the studies of electric d.c. conductivity undertaken thus far. In addition, the current status of the effort to develop an adequate capability to conduct rheological investigations is described.

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III. EXPERIMENT

The parallel plate conductance cell described previously¹⁷ was utilized in this research. This cell consists of a pair of 1.21-in. $\times$ 1.26-in. stainless-steel electrodes clamped together with a 0.05-in. gap, using pyrex spacers. A cell constant was determined for the conductance cell using a standard KCl solution.

For each test, a sample of A240 pitch was melted at about 170°C in an alumina crucible containing the conductance cell. After a period sufficient for the molten pitch to fill the gap between the cell electrodes, the sample was allowed to cool and resolidify. At this time, the programmed heating/cooling cycle was initiated. Tests were conducted at heating/cooling rates of 5°C/min, 1°C/min, and 5°C/hr to a maximum of about 450°C. In each case, the total resistance of the conductance cell was measured as a function of time (temperature). Using computer data acquisition and analysis, the results of each test were presented graphically as a plot of $\ln(k)$ as a function of $1/T$, where k is the electric d.c. conductivity and T is the absolute temperature.

Several thermogravimetric analyses (TGA) were performed to confirm the onset of volatilization of the pitch sample. TGA runs were made with a modified Perkin-Elmer TGS-1 system. In addition, optical micrographs of several samples were taken to illustrate mesophase formation.

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IV. RESULTS - D.C. CONDUCTIVITY STUDIES

A. THERMAL CYCLING EXPERIMENTS

The electrical d.c. conductivity of untreated A240 pitch is depicted as a function of absolute temperature in the form $\ln(k)$ versus $1/1000 T$ in Figs. 1-3. Nominal heating/cooling rates of 5°C/hr, 1°C/min, and 5°C/min are represented, with a maximum temperature of about 725 K (450°C). In each case, the thermal cycle is divided into three distinct regions:

1. A linear region at lower temperatures during heating, indicating an Arrhenius relation for the temperature dependence of the conductivity:

$$k = A \exp (-E/RT)$$

where the pre-exponential factor A and the activation energy E are independent of the temperature.

2. A nonlinear heating region appearing at higher temperatures, correlated with significant volatilization and mesophase formation, in which the activation energy is not independent of temperature.
3. A cooling curve which is linear over the entire temperature range.

The most striking characteristic of the conductivity curves is the apparent hysteresis effect. This will be discussed somewhat further in a later paragraph; however, it is clearly an expected behavior in view of the irreversible nature of the physical and chemical processes occurring in region 2 of the cycle.

The behavior in region 1 is comparable with that reported by Sakai, et al.¹⁸ for a series of three petroleum pitches and four coal tar pitches over the temperature range 70-160°C. These researchers observed an Arrhenius temperature dependence of the conductivity for all samples. It should be noted that in no case do any of their reported temperatures exceed the corresponding softening point of the pitch by more than about 65°C. Hence, volatilization of low molecular weight components would not be expected to exert a major influence on their results, a point which is confirmed by their report of 5% reproducibility.

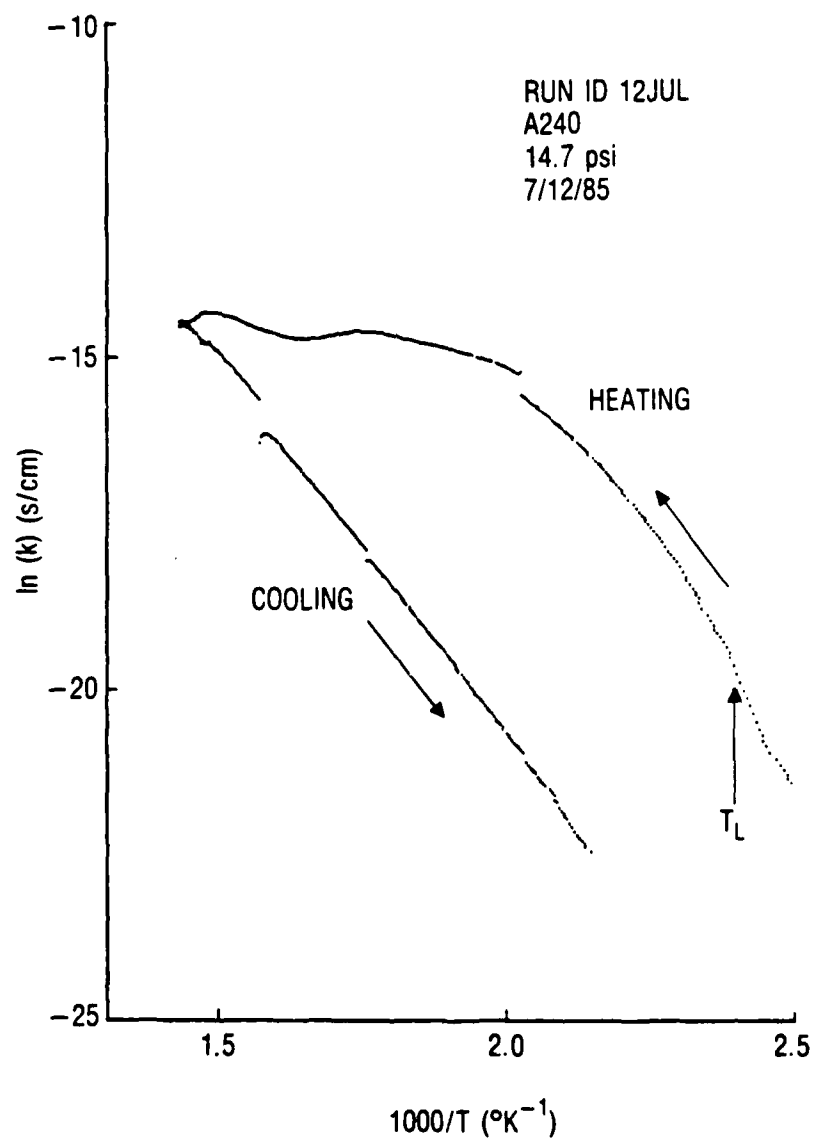


Fig. 1. D.C. conductivity: heating rate = 5°C/hr.

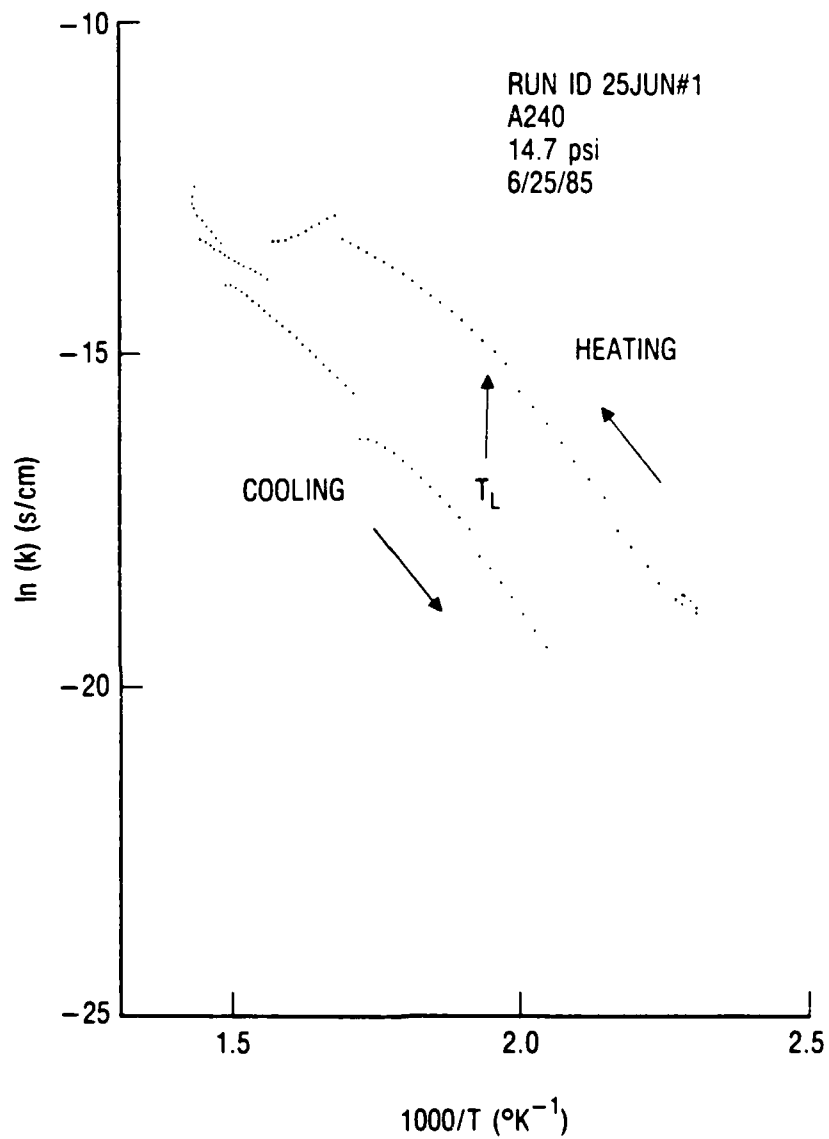


Fig. 2. D.C. conductivity: heating rate = 1°C/min.

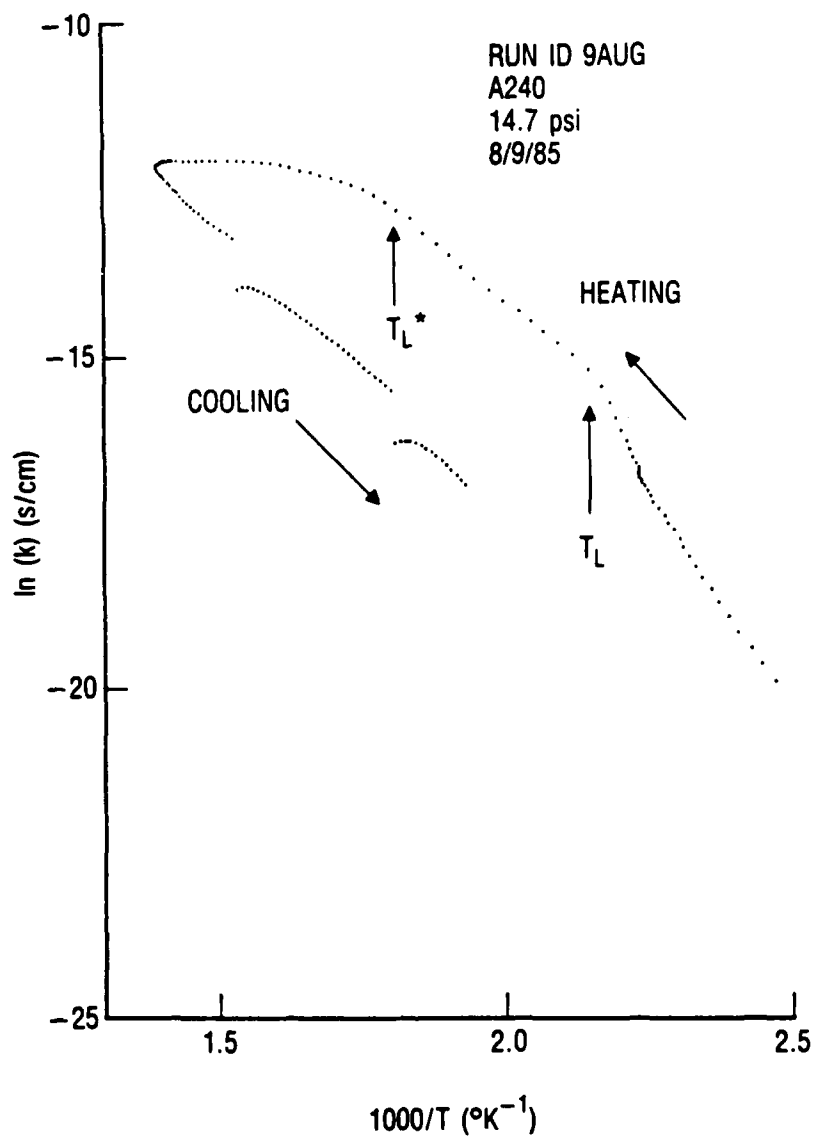


Fig. 3. D.C. conductivity: heating rate = 5°C/min.

The activation energy E_H for A240 pitch determined in the low temperature linear region (heating) of Figs. 1-3 is summarized in Table 1. E_H seems to be roughly independent of heating rate. This can be observed more directly in the figures as the slope of the curve in this region. The values of E_H are reasonably consistent with those reported by Sakai, et al.; however, they do not cite the heating rate used. In view of the low temperatures reported, their heating rate must have been relatively low.

As temperature increases, the conductivity curve displays a negative deviation from its previously linear behavior beginning at temperature T_L . Figure 4 includes TGA thermograms of A240 pitch obtained at heating rates of 0.5 and 5°C/min. Significant volatilization starts at 170 and 200°C, respectively, which correlates reasonably well with T_L from Table 1. The discrepancies can be attributed to the very large sample sizes in the conductivity measurements compared to the TGA experiments. Thus, the change in the conductivity-temperature relationship seems to be connected with the onset of volatilization of low molecular weight pitch components.

In a previous report¹⁷ it was shown that the electrical conductivity and viscosity of a Newtonian fluid in which charge transfer is ionic could be related by Walden's rule:

$$k \eta = q^2 N / 6 \pi R$$

where k is the conductivity, η is the viscosity, q is the ionic charge, N is the ionic concentration, and R is the ionic radius.

Although Walden's rule should not be expected to hold exactly, because of the many simplifying assumptions in its derivation, it could provide a qualitative indication of the conductivity vs. viscosity relation. In the temperature range associated with the onset of volatilization, pitch is rheologically Newtonian.^{9,11} Moreover, it is precisely in this temperature range (~200°C) that the viscosity-temperature relation plotted as $\ln(\eta)$ vs. $1/T$ begins a positive deviation from linearity, eventually leading to a viscosity minimum. See, for example, Figs. 18 and 22 in Evangelides and Meyer.¹⁰

Table 1. Thermal cycle electrical d.c. conductivity parameters

Run No.	Heating Rate	E_H^a kcal/mol	T_L^b K	E_C^c kcal/mol	k_m^d s/cm
9Au	5°C/min	26	475 ^e (200°C)	18	6×10^{-6}
25JU1	1°C/min	23	520 (250°C)	22	4×10^{-6}
300	5°C/hr	36	420 (160°C)	23	5×10^{-7}
12JUL	5°C/hr	34	420 (160°C)	23	6×10^{-7}

Notes:

- Region 1 (heating) activation energy.
- Temperature at which deviation from Arrhenius relation occurs during heating, i.e., onset of volatilization.
- Region 3 (cooling) activation energy.
- Approximate maximum electric d.c. conductivity.
- It is believed that at this, the highest heating rate, significant volatilization actually commences at $T_L^* = 280^\circ\text{C}$. See Fig. 3.

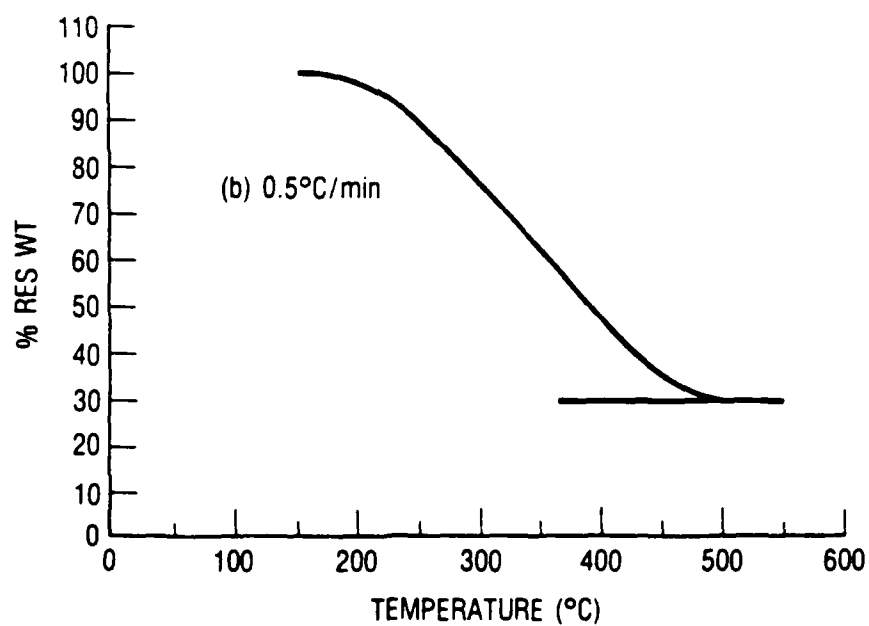
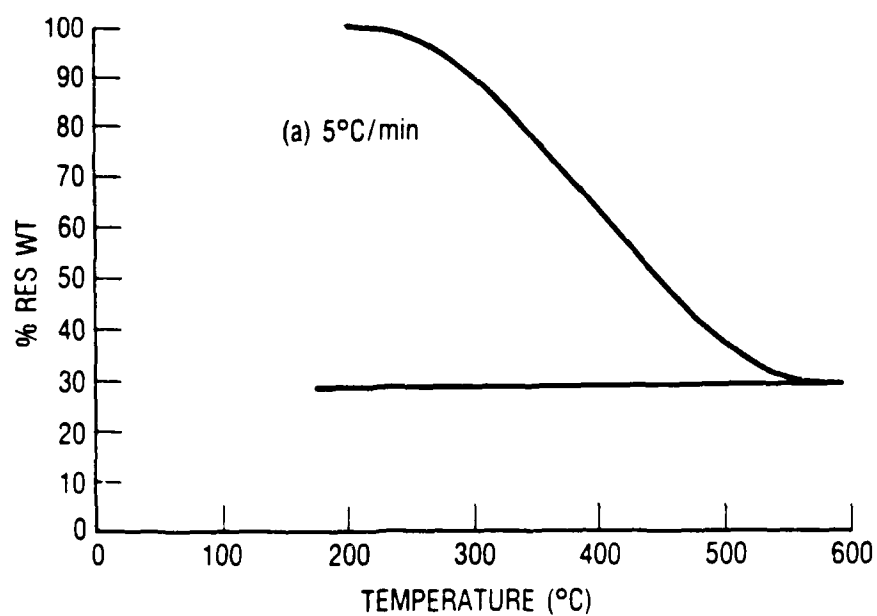


Fig. 4. TGA thermograms: A240 pitch.

The negative deviation of the conductivity curve is consistent with the positive deviation of the viscosity curve and Walden's rule where $k \sim \eta^{-1}$. As pointed out by Evangelides and Meyer, the viscosity behavior indicates that some polymerization, or at least molecular ordering, occurs in this temperature region even if mesophase formation is not significant. An increase in average molecular size would result in reduced ion mobility and reduced conductivity.

At this point it must be noted that the effect of heating rate on T_L as indicated in Table 1 is not entirely consistent with the above interpretation. That is, T_L should increase with increasing heating rate. Increased heating rate leads to a decrease in total volatilization in a given time. Hence, the observation of volatilization is delayed in time to higher temperature. This does not appear to be the case for the run at 5°C/min whose T_L is less than that for 1°C/min. On the other hand, note in Fig. 3 (5°C/min), the presence of a second (approximately) linear region, separated from the first by a "knee," the location of which was taken as T_L . If this knee is interpreted as spurious, or at least as resulting from some mechanism other than volatilization, then T_L^* represents the commencement of significant volatilization. The large sample size in conjunction with the high heating rate could also be responsible for this behavior. With this interpretation, since $T_L^* = 280^\circ\text{C}$, the data fall into place.

Referring again to Figs. 1-3, continued heating causes the electrical conductivity to deviate more markedly from its original Arrhenius relationship. Eventually conductivity reaches a maximum with increasing temperature. Recall that the conductivity curves are dynamic curves. That is, the time necessary to reach any specific temperature depends upon the heating rate. Thus, the amount of volatilization and the extent of mesophase formation at the maximum temperature differ for the three heating rates.

Reasonably enough, therefore, the apparent electrical conductivity maximum increases with heating rate. This is also shown in Table 1. In addition, the conductivity during cooling is lower, at any temperature, than during the heating portion of the cycle, because of the irreversible changes that occur during the heating. This leads to the characteristic hysteresis of the cycle.

Perhaps the most striking feature of the conductivity curves is the cooling portion of the cycle (region 3). Figures 1-3 show that the behavior of the conductivity has returned to an Arrhenius relation. Moreover, the activation energy for the cooling process is independent of cooling rate, and it is virtually the same activation energy observed during heating in region 1.

These observations argue for the conclusion that the conductivity is almost entirely due to mobility of the lower molecular weight ionic components in the isotropic pitch fraction of the sample. As the concentration of these species decreases due to volatilization and their average size increases due to polymerization, the conductivity decreases in competition with the normal viscosity-temperature effect. This seems reasonable as long as the mesophase is the dispersed phase and the isotropic component acts as the continuous phase. At the phase inversion point, viscosity undergoes a local maximum.⁹ Similar behavior should be expected of conductivity in this region once the mesophase becomes the continuous phase in which are dispersed regions of isotropic pitch. No experiments have as yet been performed to specifically address this point.

Figure 5 represents the behavior of a sample of untreated A240 pitch subjected to repeated thermal cycles to 450°C. Figure 5(a) shows the initial cycle, while (b) and (c) represent the second and third cycles, respectively. The substantial hysteresis observed in the first cycle has virtually disappeared by the second cycle. Deviations from linearity are observed in cycles 2 and 3, but the heating process is virtually reversed on cooling, and no hysteresis is observed. It would seem from this behavior that the deviation from linearity is not due solely to volatilization and polymerization, since a hysteresis effect would follow. It is likely that at these temperatures a simple Arrhenius relation with a single activation energy is simply no longer adequate to represent the conduction process, even in the absence of volatilization. To determine whether mesophase formation was proceeding rapidly enough to cause this phenomenon, a fourth cycle (not shown) was halted at its maximum temperature (450°C) for 10 hr. No significant change in d.c. conductivity was observed during this period.

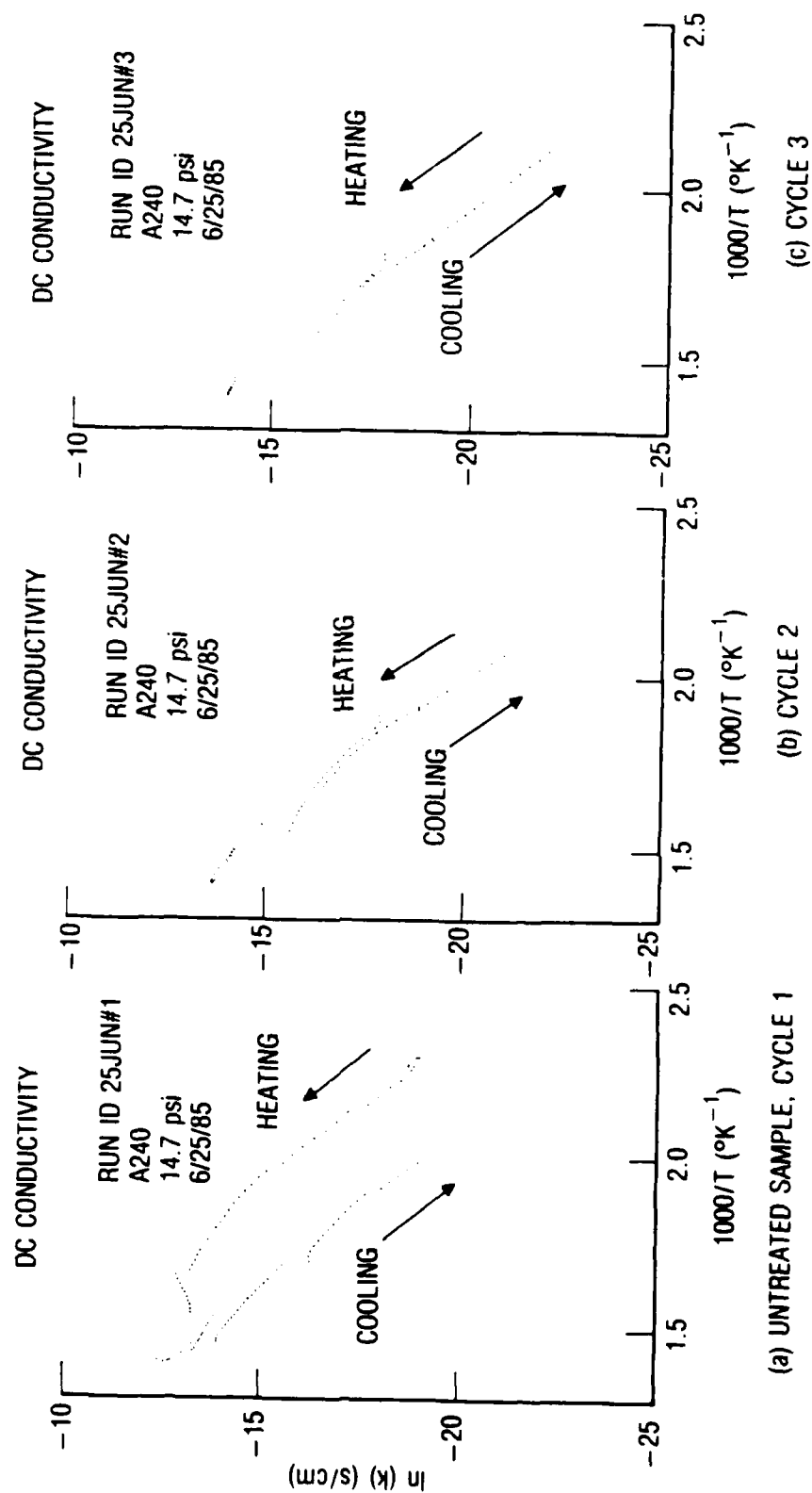


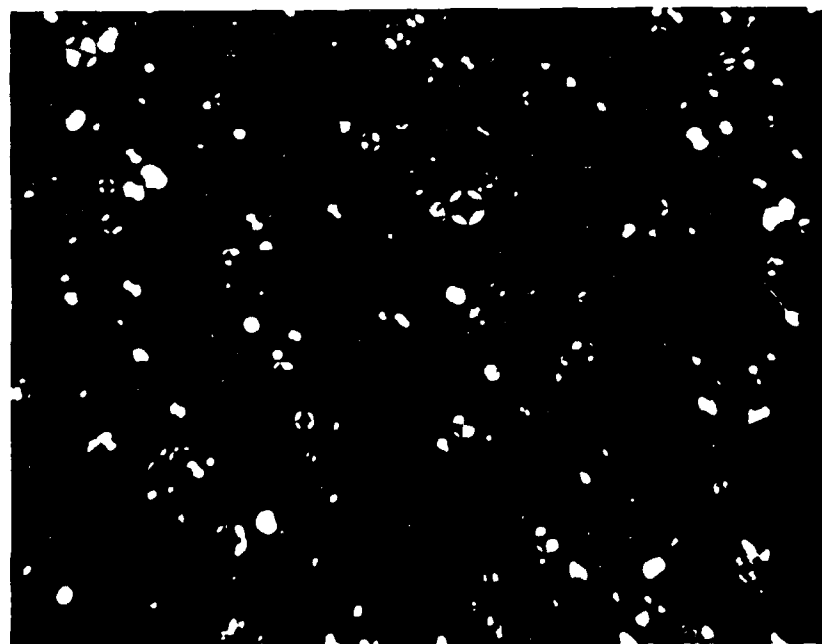
Fig. 5. Thermal cycles.

Optical micrographic analysis of material taken from the cell at the conclusion of this experiment revealed a substantial amount of mesophase formation within the sample. Figure 6 shows micrographs of bulk material taken from the bottom of the cell as well as from the inter-electrode region. Mesophase formation proceeds slowly at lower temperature. Therefore, it seems logical to conclude that most of the mesophase in the sample was formed during the last 10 hr of the run under isothermal conditions (450°C). It is significant that no change in electrical d.c. conductivity was observed during this period.

B. REPRODUCIBILITY OF ELECTRICAL CONDUCTIVITY CURVES

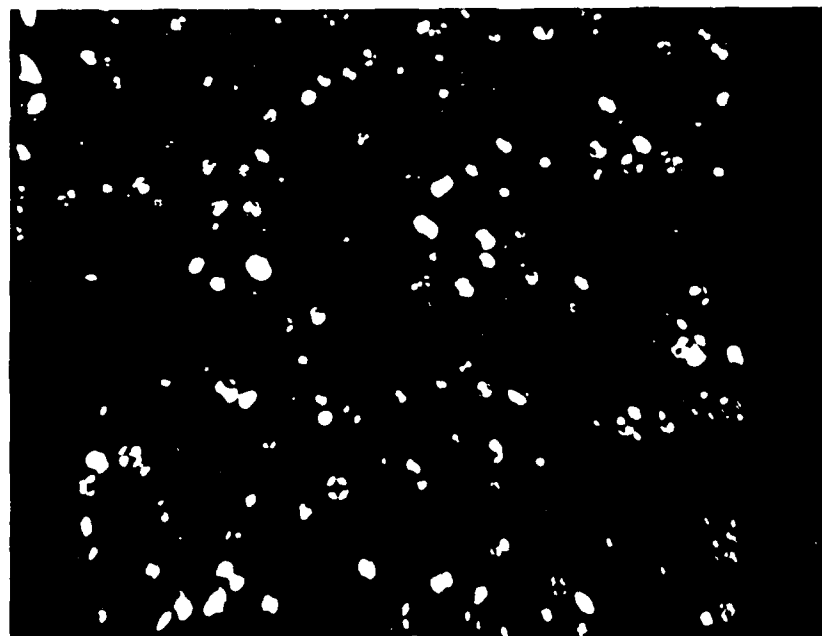
Figures 7 and 8 illustrate the extent to which the conductivity cycles may be considered reproducible. Illustrated in Fig. 7 are two runs taken at 5°C/hr with untreated pitch. Figure 8 depicts two runs at 1°C/min with pitch samples which had been preheated to 450°C under identical conditions. Good agreement is exhibited in both cases.

Also illustrated in these figures, as well as in others of the conductivity curves, is the presence of substantial discontinuities. It is thought that these result from the formation and collapse of vapor bubbles in the narrow gap between the plates of the conductivity cell. This argument is supported by the observation that the conductivity curve usually returns to a position reasonably consistent with the trend prior to the discontinuity. Several such occurrences are indicated by arrows in Figs. 7 and 8.



x 100

(a) BULK MATERIAL



x 100

(b) INTER-ELECTRODE MATERIAL

Fig. 6. Optical micrographs of A240 pitch (run 25 JU 4)

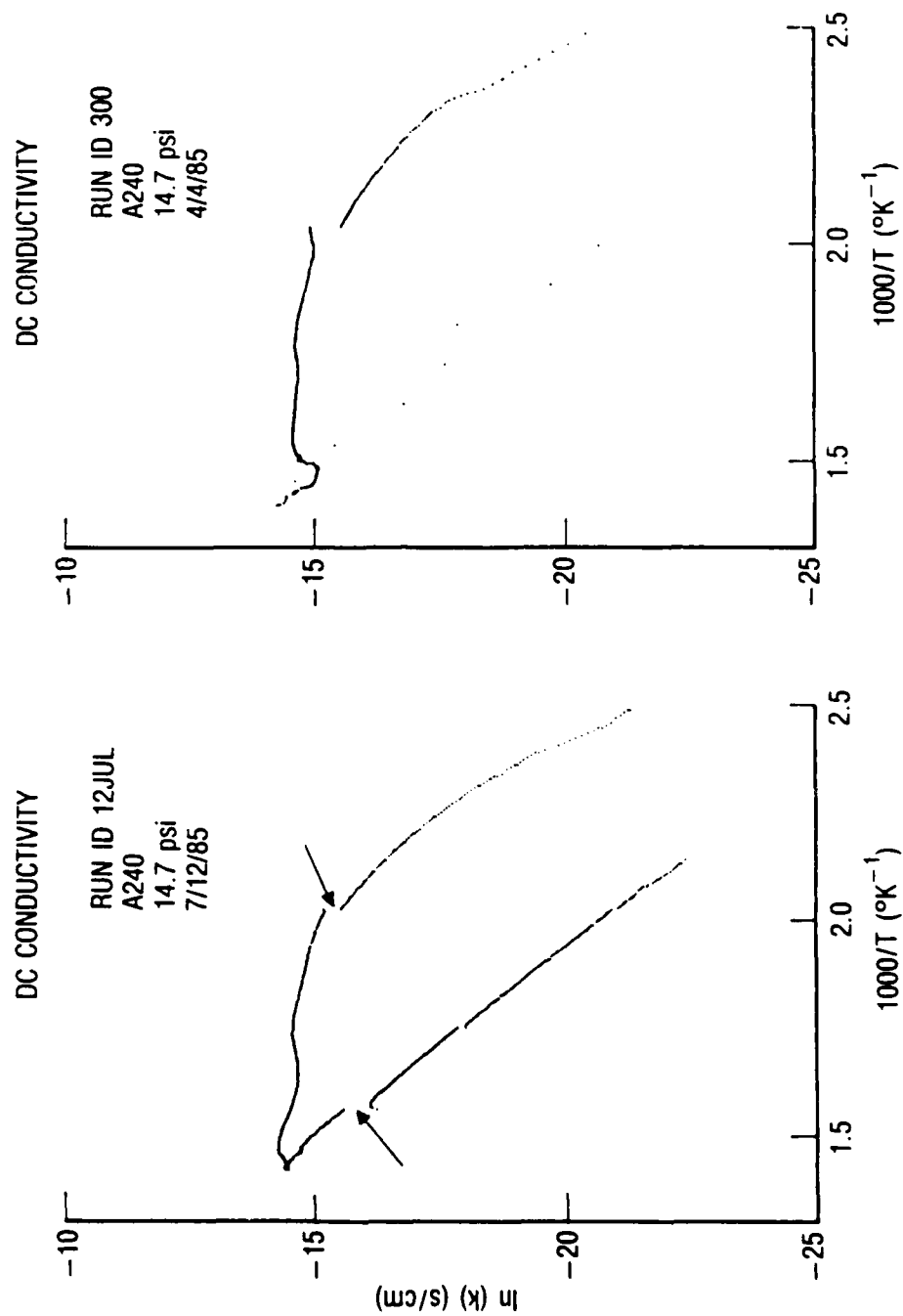


Fig. 7. Reproducibility of results: heating rate = $5^{\circ}C/hr$.

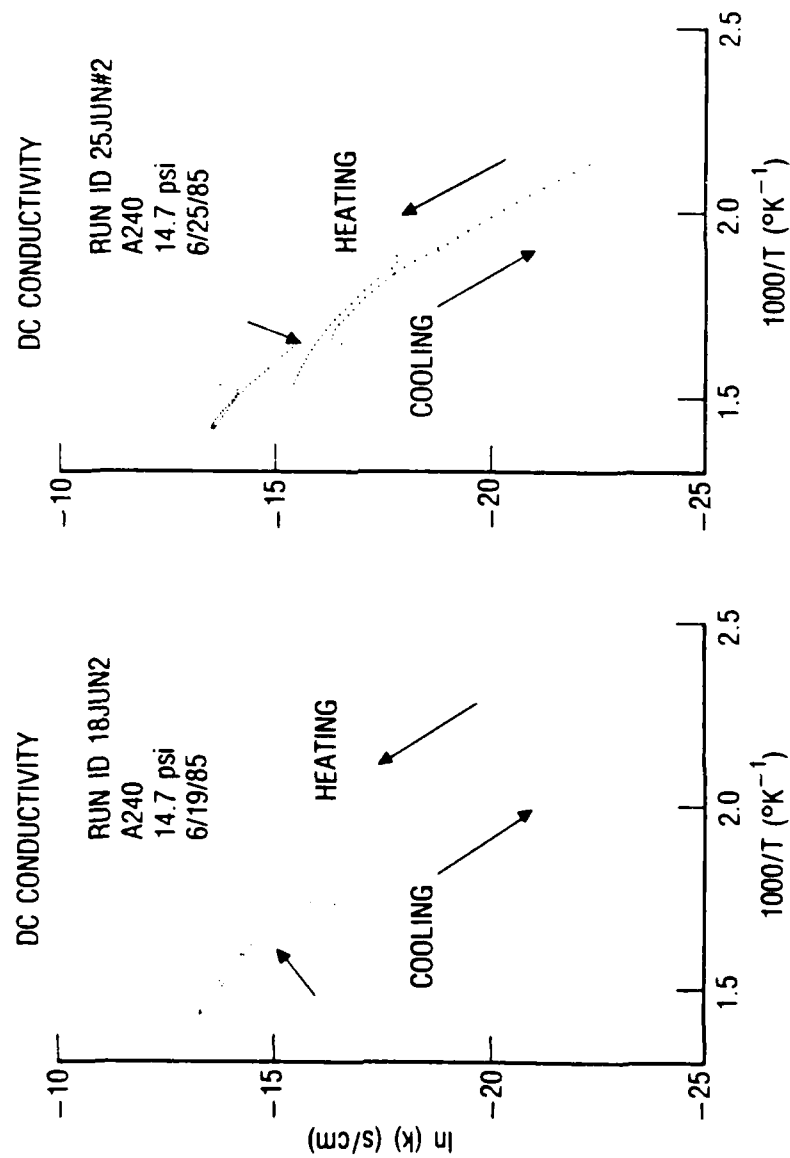


Fig. 8. Reproducibility of results: samples preheated to 450°C; heating rate = 1°C/min.

V. RESULTS - PROGRESS ON VISCOSITY INSTRUMENTATION

The high-temperature viscometer was received roughly at the end of the period covered by this report. This instrument is comprised of a Haake ROTOVISCO RV2 rotating viscometer and a Theta furnace and controller capable of temperatures reaching 1000°C.

Effort has proceeded to complete the installation of this system. Of primary concern is the design of an atmospheric control system for the viscometer. This, of course, is necessary for the protection of laboratory personnel as well as the sensitive measuring heads of the viscometer. These must be kept free of condensible vapors encountered in pitch work.

The National Bureau of Standards viscosity calibration standards have been obtained. These will be used to establish the absolute viscosity range accessible to the system and to calibrate the viscometer.

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VI. CONCLUSIONS AND RECOMMENDATIONS

The temperature dependence of the d.c. conductivity of molten pitch is strongly influenced by the volatilization and polymerization of low molecular weight pitch components. The role of mesophase formation on this property, however, remains uncertain. Results thus far indicate that conductivity may not be sensitive to mesophase formation, at least in an isothermal environment.

It is recommended that in the immediate term attention first be directed at investigating the influence of mesophase formation on the viscosity of pitch. The objective of this study should be to correlate viscometric properties to mesophase content. Because of the complex rheological nature of pitch, this may necessitate the consideration of non-Newtonian, viscoelastic and time-dependent phenomena.

Further attention to the measurement of d.c. conductivity is warranted to settle the question of how mesophase content influences this property. To this end, it is suggested that isothermal tests in which mesophase content is carefully monitored should be most appropriate. High-pressure d.c. conductivity measurements should not be undertaken until results clearly indicate an influence of mesophase content on conductivity.

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