POLARIZED TRANSIENT HOT WIRE THERMAL CONDUCTIVITY MEASUREMENTS[*]

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ABSTRACT

Additional experimental uncertainty is introduced in thermal conductivity data obtained with the transient hot-wire technique when bare hot wires are used in polar liquids. The use of a dc polarization voltage applied between the hot wires and the cell wall greatly reduces this uncertainty for polar liquids. Differences between polarized and nonpolarized experiments are described for the alternative refrigerants 1,1,1,2-tetrafluoroethane (R134a) and 1-chloro-1,1-difluoroethane (R142b). Comparisons are made between data from the polarized transient hot-wire technique and other experimental techniques for R142b. The polarization technique enables existing transient hot-wire instruments with bare wires to study the thermal conductivity of moderately polar liquids with confidence.

INTRODUCTION

The transient hot-wire technique is well proven for nonpolar liquids and gases. In this technique the hot wires are immersed in the fluid of interest and are used as electrical heat sources and resistance thermometers. There is generally no electrical insulation on the hot wires, and problems arise if the fluid has significant electrical conductivity. This is the case for polar liquids such as water or hydrochlorofluoro-

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carbon refrigerants. Extremely pure polar liquids do not normally exhibit significant electrical conductivity. However, polar liquids can easily dissolve enough ionic material to become electrically conducting if they are stored in metallic containers.

Several phenomena are observed when a transient hot-wire instrument is filled with a polar liquid, such as R134a or R142b. First, the electrical resistance between the hot wires and the cell walls degrades. This resistance is normally over 40 MΩ when nonpolar liquids are present in the cells. The resistance drops to about 5 MΩ when either liquid R134a or R142b is present in our hot wire cells. The electrical isolation degrades with elapsed time after the cell is filled, even after repeated fillings. The electrical conductivity of the fluid also increases with the concentration of water in the refrigerant sample. The liquid refrigerants appear to be dissolving some ionic material from our hot-wire cells. The quantity of ionic material which is soluble seems to increase with the water concentration in the sample. When there is measurable electrical conduction in the fluid there is also an electrical potential present, between the hot wires and the cell walls, which is of the order of 50 mV.

There are two possible solutions to this polar liquid problem. First, extremely pure liquids could be measured in an environment where they cannot dissolve ionic impurities. Alternatively, experimental techniques which can tolerate the presence of dilute ionic impurities in the fluid could be used. Because of the problems encountered with obtaining and storing extremely pure polar liquids, the development of experimental techniques which can be used in the presence of dilute ionic impurities is the favored solution. The most obvious modification of the transient hot-wire technique is to provide electrical insulation between the surface of the hot wires and the fluid. Insulated hot wires can be used with both polar liquids and highly ionic solutions. In most cases, tantalum wires are electrolytically coated with an insulating layer of tantalum pentoxide (Alloush et al., 1982; Wakeham and Zalaf, 1986). The use of platinum hot wires coated with a polyester film is also described (Nagasaka and Nagashima, 1981). All of these techniques are relatively difficult and have more experimental uncertainty due to addition of the electrical-insulation layer.

Two modifications to the transient hot-wire technique allow bare hot wires to be used in polar liquids. Transient hot-wire measurements have been made on deionized water using an ac drive voltage to heat bare hot wires and monitor their resistance change (Dietz et al., 1981). The electrical conductivity of the water in the cell degraded to 10^{-6} $\Omega^{-1}cm^{-1}$ upon filling the metallic hot-wire cell (Dietz et al., 1981). The 20 kHz frequency of the ac drive voltage assured that there was no migration of the ionic impurities in the alternating electric field around the hot wires. Alternatively, dc polarization voltages have been applied between bare platinum hot wires and the cell wall (Nieto de Castro et al., 1989; Ross et al., 1990). The permanent dc polarization voltage establishes compact double layers near the surfaces in the resulting electric field (Bard and Faulkner, 1980). These double layers contain solvated ions, with a charge which is opposite that of the metallic surface which they surround, in order to maintain charge neutrality in the solution. The compact double layers effectively shield the ions in the bulk solution from the charges which are

present on the metallic surfaces in the cell during an experiment. Since the compact double layers are established before the heating voltage is applied, their presence does not introduce errors in the measurement of the wire resistance change and hence the measured temperature rise. This work examines discrepancies between polarized and nonpolarized transient hot-wire measurements of the thermal conductivity of polar liquids.

EXPERIMENTAL

The measurements reported in this work were obtained with two transient hot-wire instruments which have been previously described. The low temperature instrument (Roder, 1981) operates from 30 to 330 K at pressures to 70 MPa. The thermal conductivity cell is constructed of a beryllium-copper alloy and uses bare platinum hot wires. The high temperature instrument (Perkins et al., 1991) operates from 300 to 750 K at pressures to 70 MPa. This thermal conductivity cell is constructed of type-316 stainless steel and also uses bare platinum wires. These instruments use 12.7 μm diameter platinum wires as transient electrical heat sources and as resistance thermometers. The electrical insulation on the leads inside the cells is provided by polytetrafluoroethane, polyimide, and polyester. A typical experiment is 1 s in duration with a final temperature rise of 1 to 4 K. During a 1 s experiment, the temperature rise is recorded every 4 ms for a total of 250 data points. The instruments are designed to approximate the transient line source model as closely as possible. Deviations from this model are handled as corrections. Ideally the wire temperature rise, ΔT, is given as a function of elapsed time t by

$$\Delta T = \frac{q}{4\pi\lambda} \ln\left(\frac{4at}{r_o^2 C}\right) = \frac{q}{4\pi\lambda} \ln\left(\frac{4a}{r_o^2 C}\right) + \frac{q}{4\pi\lambda} \ln(t), \tag{1}$$

where q is the power input per unit length of wire, r_o is the radius of the wire, λ is the thermal conductivity, $a=\lambda/\rho C_p$ is the thermal diffusivity, ρ is the density, C_p is the isobaric heat capacity, and $C=1.781...$ is the exponential of Euler's constant. As seen in Eqn. (1), the fluid's thermal conductivity can be found as the slope of the line through a plot of the temperature rise versus the logarithm of elapsed time. For measurements of the thermal conductivity of nonpolar fluids the repeatability of both instruments is ±0.3% with an uncertainty less than ±1.0% (Roder, 1981; Perkins et al. 1991).

For the polarized experiments the grounding arrangement for the hot-wire cells is modified as shown in Figure 1. The cells are now biased relative to ground using adjustable dc power supplies. In the presence of an electric field an electric double layer is formed near the surface of the hot wires. In electrode polarization experiments this double layer is characterized by an equivalent resistor and capacitor in series, or an RC time constant. This RC time constant is a function of the magnitude of the polarization voltage, described by models such as the Gouy-
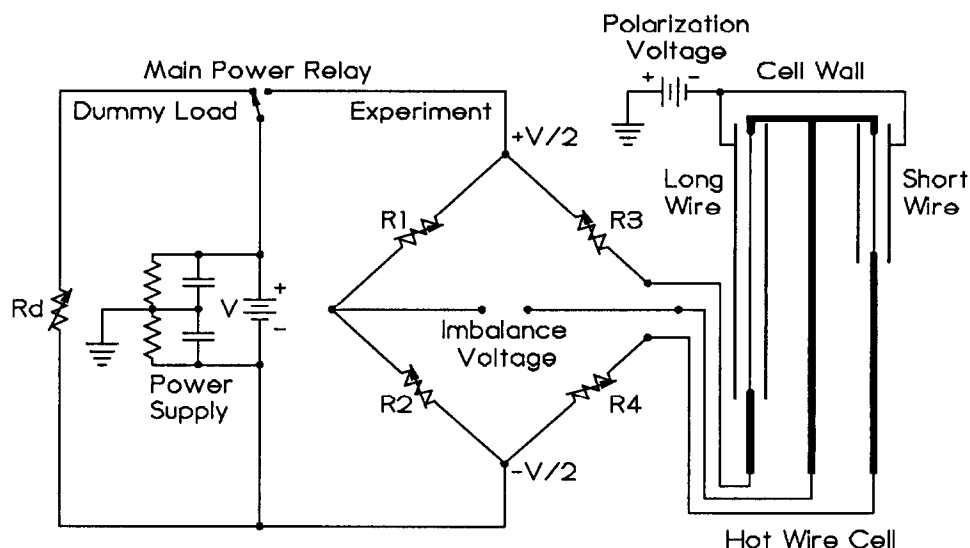
Fig. 1. Application of the polarization voltage between the hot wires and the cell walls.

Chapman-Stern model (Bard and Faulkner, 1980), until the compact double layer is fully established. When the compact double layer is established, its capacitance is constant and application of the experiment heating voltage does not result in changes in the double layer structure near the surface of the hot wires. The compact double layer shields the hot wires from any ions in the bulk fluid when the experimental heating voltage is applied.

LIQUID REFRIGERANT DATA

A selected set of thermal conductivity data is presented in this work to demonstrate the performance of the polarized transient hot-wire technique for liquid refrigerant measurements. In the tables, T_0 is the initial cell temperature and T is the average temperature for a given experiment. The data in Table 1 explore the effect of the magnitude of the polarization voltage on the thermal conductivity measurement for R134a near 300 K. The data in Table 2 contrast the effect of temperature on transient hot-wire thermal conductivity measurements of R134a, with and without a polarization voltage. Finally, we need to show that the polarized transient hot-wire measurements of refrigerants are in good agreement with independent measurements using another experimental technique. For this verification we have selected R142b, since it has recently been studied by Sousa et al. (1991) using a steady-state concentric-cylinder instrument with an experimental uncertainty of ±2%. Thermal conductivity data are provided for R142b near 300 K as a function of density in Table 3, with and without a polarization voltage. The densities reported in Tables 1, 2, and 3 are calculated using the corresponding states formulation of Huber and Ely (1991).

TABLE 1

Thermal conductivity data for saturated liquid R134a with increasing polarization voltage.

T_o (K)	V_{pol} (V)	T (K)	P (MPa)	ρ (mol L^{-1})	λ (W m^{-1}K^{-1})
299.530	0.00	303.276	0.6974	11.632	0.08097
299.534	0.00	302.544	0.6974	11.660	0.08150
299.538	0.00	301.904	0.6974	11.684	0.08222
299.532	0.00	301.342	0.6974	11.706	0.08311
299.506	2.00	303.252	0.6966	11.633	0.08100
299.513	2.00	302.538	0.6966	11.661	0.08127
299.506	2.00	301.887	0.6966	11.685	0.08230
299.508	2.00	301.312	0.6966	11.707	0.08270
299.498	4.00	303.215	0.6965	11.635	0.08108
299.492	4.00	302.492	0.6965	11.662	0.08181
299.497	4.00	301.853	0.6965	11.686	0.08210
299.495	4.00	301.291	0.6965	11.708	0.08211
299.466	6.00	303.192	0.6958	11.636	0.08090
299.461	6.00	302.480	0.6958	11.663	0.08131
299.465	6.00	301.849	0.6958	11.687	0.08171
299.466	6.00	301.295	0.6958	11.707	0.08219
299.494	8.00	303.231	0.6945	11.634	0.08065
299.501	8.00	302.505	0.6945	11.662	0.08137
299.496	8.00	301.868	0.6945	11.686	0.08173
299.500	8.00	301.293	0.6945	11.707	0.08217
299.478	10.00	303.213	0.6945	11.635	0.08090
299.477	10.00	302.502	0.6945	11.662	0.08129
299.483	10.00	301.883	0.6945	11.685	0.08155
299.491	10.00	301.313	0.6945	11.707	0.08223
299.467	12.00	303.177	0.6938	11.636	0.08090
299.460	12.00	302.461	0.6938	11.663	0.08149
299.459	12.00	301.831	0.6938	11.687	0.08174
299.461	12.00	301.260	0.6938	11.709	0.08210

TABLE 2

Thermal conductivity data for R134a near the saturated liquid line.

T_o (K)	V_{pol} (V)	T (K)	P (MPa)	ρ (mol L^{-1})	λ (W m^{-1}K^{-1})
200.868	0.00	203.940	2.2338	14.701	0.12593
200.869	0.00	203.542	2.2352	14.711	0.12626
200.871	0.00	203.167	2.2373	14.721	0.12678
200.870	0.00	202.826	2.2373	14.730	0.12720
201.187	6.00	204.245	2.2815	14.693	0.12571
201.188	6.00	203.845	2.2991	14.704	0.12596
201.188	6.00	203.471	2.3216	14.715	0.12647
201.187	6.00	203.127	2.3427	14.724	0.12680
219.680	0.00	222.827	1.2919	14.176	0.11735
219.682	0.00	222.388	1.2947	14.188	0.11782
219.682	0.00	221.982	1.2933	14.199	0.11814

TABLE 2 (continued)

T_o (K)	V_{pol} (V)	T (K)	P (MPa)	ρ (mol L^{-1})	λ (W m^{-1}K^{-1})
219.683	0.00	221.610	1.2919	14.209	0.11878
219.675	6.00	222.835	1.2792	14.175	0.11690
219.675	6.00	222.392	1.2757	14.187	0.11730
219.677	6.00	221.994	1.2750	14.198	0.11757
219.675	6.00	221.616	1.2757	14.208	0.11795
239.030	0.00	242.169	1.3183	13.639	0.10790
239.031	0.00	241.693	1.3183	13.653	0.10902
239.028	0.00	241.259	1.3183	13.665	0.11051
239.027	0.00	240.870	1.3183	13.676	0.11078
239.069	6.00	242.265	1.3201	13.637	0.10796
239.069	6.00	241.786	1.3201	13.650	0.10837
239.069	6.00	241.346	1.3250	13.663	0.10880
239.067	6.00	240.950	1.3250	13.674	0.10912
260.309	0.00	264.067	2.1011	13.023	0.09879
260.310	0.00	263.504	2.1054	13.040	0.09931
260.313	0.00	262.990	2.1054	13.056	0.09997
260.309	0.00	262.511	2.1110	13.070	0.10039
260.675	6.00	264.421	2.0876	13.012	0.09808
260.673	6.00	263.854	2.0855	13.029	0.09847
260.674	6.00	263.336	2.0749	13.044	0.09886
260.675	6.00	262.868	2.0749	13.058	0.09899
280.253	0.00	284.017	1.9268	12.388	0.08997
280.250	0.00	283.414	1.9268	12.408	0.09065
280.248	0.00	282.858	1.9303	12.426	0.09125
280.247	0.00	282.353	1.9303	12.443	0.09201
280.345	6.00	284.108	1.6697	12.374	0.08873
280.348	6.00	283.500	1.6767	12.394	0.08921
280.347	6.00	282.947	1.6873	12.413	0.08969
280.343	6.00	282.446	1.6964	12.430	0.08995
299.558	0.00	303.046	5.1772	11.901	0.08445
299.556	0.00	302.538	5.1744	11.918	0.08473
299.554	0.00	302.075	5.1744	11.934	0.08506
299.554	0.00	301.649	5.1744	11.948	0.08578
299.510	6.00	303.007	5.1459	11.900	0.08325
299.510	6.00	302.505	5.1494	11.918	0.08312
299.506	6.00	302.041	5.1515	11.934	0.08378
299.503	6.00	301.614	5.1543	11.948	0.08363

ANALYSIS

The effect of the electric double layer on a thermal conductivity measurement is shown in Figure 2. Figure 2a shows that there is significant curvature relative to the full heat transfer model when there is no polarization voltage. Similar curvature is expected if there is a significant thermal radiation correction (Perkins et al., 1991). If this curvature is due to radiation or to some other parasitic heat transfer mechanism,

TABLE 3

Thermal conductivity data for R142b near 300 K as a function of density.

T_0 (K)	V_{pol} (V)	T (K)	P (MPa)	ρ (mol L^{-1})	λ (W m^{-1}K^{-1})
300.119	0.00	303.933	69.5827	12.421	0.11119
300.114	0.00	303.374	69.5778	12.429	0.11079
300.112	0.00	302.846	69.5757	12.436	0.11163
300.107	0.00	302.404	69.5722	12.442	0.11153
300.111	0.00	304.064	56.9517	12.236	0.10725
300.106	0.00	303.488	56.9531	12.244	0.10659
300.107	0.00	302.939	56.9517	12.252	0.10681
300.102	0.00	302.464	56.9517	12.259	0.10749
300.090	0.00	304.184	43.8297	12.017	0.10185
300.090	0.00	303.578	43.8269	12.026	0.10130
300.087	0.00	303.017	43.8241	12.035	0.10097
300.087	0.00	302.512	43.8227	12.043	0.10234
300.084	0.00	304.346	31.9747	11.788	0.09649
300.084	0.00	303.726	31.9628	11.798	0.09575
300.082	0.00	303.145	31.9593	11.808	0.09695
300.079	0.00	302.615	31.9550	11.816	0.09717
300.071	0.00	303.868	20.8523	11.545	0.09105
300.073	0.00	303.266	20.8552	11.556	0.09105
300.067	0.00	302.705	20.8552	11.566	0.09211
300.063	0.00	302.201	20.8495	11.576	0.09325
300.083	0.00	303.979	14.0356	11.361	0.08737
300.083	0.00	303.346	14.0370	11.374	0.08831
300.079	0.00	302.783	14.0370	11.385	0.08867
300.077	0.00	302.264	14.0370	11.396	0.08998
300.088	0.00	304.129	7.2003	11.145	0.08394
300.090	0.00	303.478	7.2003	11.160	0.08482
300.089	0.00	302.887	7.1975	11.173	0.08483
300.087	0.00	302.350	7.1961	11.185	0.08589
300.095	0.00	304.267	1.6446	10.934	0.08064
300.090	0.00	303.589	1.6425	10.951	0.08103
300.087	0.00	302.971	1.6425	10.967	0.08103
300.087	0.00	302.416	1.6425	10.981	0.08207
300.063	6.00	303.921	67.2608	12.389	0.10786
300.058	6.00	303.360	67.2496	12.397	0.10806
300.063	6.00	302.825	67.2390	12.404	0.10816
300.058	6.00	302.378	67.2327	12.410	0.10798
300.079	6.00	304.058	56.5867	12.230	0.10427
300.075	6.00	303.464	56.5831	12.239	0.10436
300.070	6.00	302.932	56.5817	12.246	0.10458
300.070	6.00	302.445	56.5824	12.253	0.10421
300.083	6.00	304.178	44.0162	12.021	0.09987
300.095	6.00	303.581	44.0218	12.030	0.09975
300.096	6.00	303.029	44.0085	12.038	0.09993
300.090	6.00	302.526	44.0042	12.046	0.09991
300.033	6.00	304.297	32.0776	11.791	0.09501
300.031	6.00	303.656	32.0776	11.801	0.09495
300.028	6.00	303.082	32.0762	11.811	0.09483
300.024	6.00	302.560	32.0762	11.820	0.09507
300.068	6.00	303.839	22.3118	11.581	0.09066

TABLE 3 (Continued)

T_o (K)	$\dot{V}_{pol}$ (V)	T (K)	P (MPa)	ρ (mol L^{-1})	λ (W m^{-1}K^{-1})
300.063	6.00	303.253	22.3118	11.592	0.09064
300.063	6.00	302.704	22.3118	11.602	0.09083
300.063	6.00	302.212	22.3118	11.611	0.09124
300.068	6.00	303.975	13.9786	11.360	0.08703
300.067	6.00	303.344	13.9772	11.372	0.08677
300.065	6.00	302.779	13.9793	11.384	0.08725
300.064	6.00	302.272	13.9793	11.394	0.08732
300.056	6.00	304.101	7.1833	11.145	0.08375
300.055	6.00	303.447	7.1855	11.160	0.08368
300.052	6.00	302.865	7.1890	11.173	0.08353
300.050	6.00	302.321	7.1728	11.185	0.08392
300.064	6.00	304.235	1.4614	10.928	0.08009
300.065	6.00	303.564	1.4642	10.945	0.08040
300.062	6.00	302.959	1.4656	10.960	0.08003
300.059	6.00	302.423	1.4670	10.973	0.08022

it will not be altered by the presence of the polarization voltage. Figure 2b shows that the curvature is eliminated when a dc polarization voltage of 6 V is present, so the curvature is due to polarization of the fluid and not to parasitic heat transfer. It remains to determine how much polarization voltage is necessary and whether too much is detrimental.

Polarization voltage

Deviations in the thermal conductivity results from Table 1 are shown in Figure 3 as a function of experiment temperature rise (T-T_o) for seven different polarization voltages. The baseline in Figure 3 is the mean of the data for the experiments with polarizations from 6 V to 12 V. For experiments with polarization voltages of 6 V and larger the repeatability with respect to applied power is ±0.3% which is consistent with observations for nonpolar fluids (Perkins et al., 1991). The experiments with no polarization voltage yield thermal conductivity data which are systematically high. This trend becomes more pronounced for small temperature rises, with 1.8 K temperature rise experiments generating thermal conductivity data which is 1.4% high. For R134a measurements with no polarization voltage the repeatability with respect to applied power is about ±1%.

Temperature dependence

Deviations in the thermal conductivity data of Table 2 for R134a near the saturated liquid line are shown in Figure 4 as a function of temperature. The baseline of Figure 4 is the saturated liquid thermal conductivity fit for R134a given by
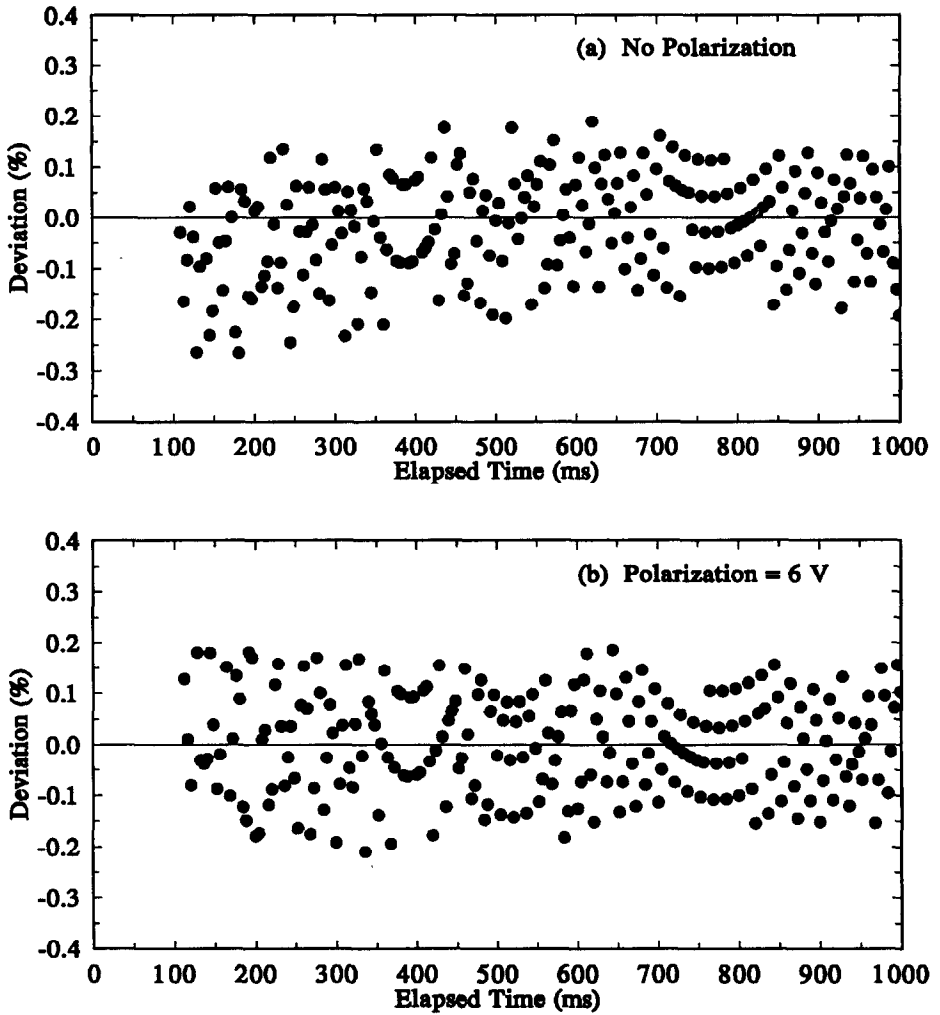
Fig. 2. Deviations between the experimental temperature rise and the heat transfer model for saturated liquid R134a at 300 K with temperature rises of 1.8 K: (a) No polarization (b) Polarization = 6 V.

$$\lambda_{sl} = 2.2029 \times 10^{-1} - 4.6779 \times 10^{-4} \ T + 1.2118 \times 10^{-8} \ T^2, \tag{2}$$

where the thermal conductivity is in W m^{-1} K^{-1}, and T can range from 240 to 360 K. Figure 4 shows that the scatter for the polarized experiments is ±0.3% and that these agree well with Eqn. (2). The experiments with no polarization have an increased scatter of ±1% which is attributed to the power dependence shown in Figure 3 for
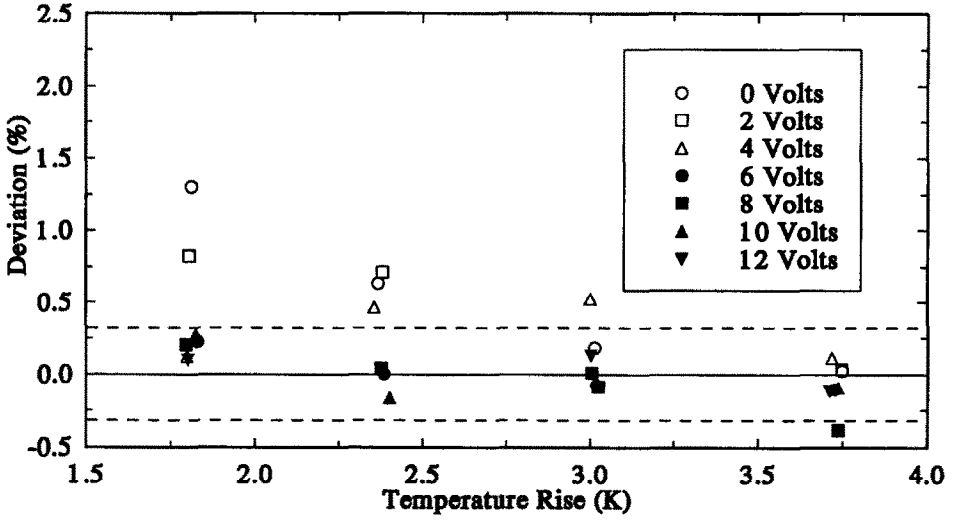
Fig. 3. Scatter in the measured thermal conductivity of saturated liquid R134a as a function of the experiment temperature rise (T-T$_s$) for various polarization voltages.
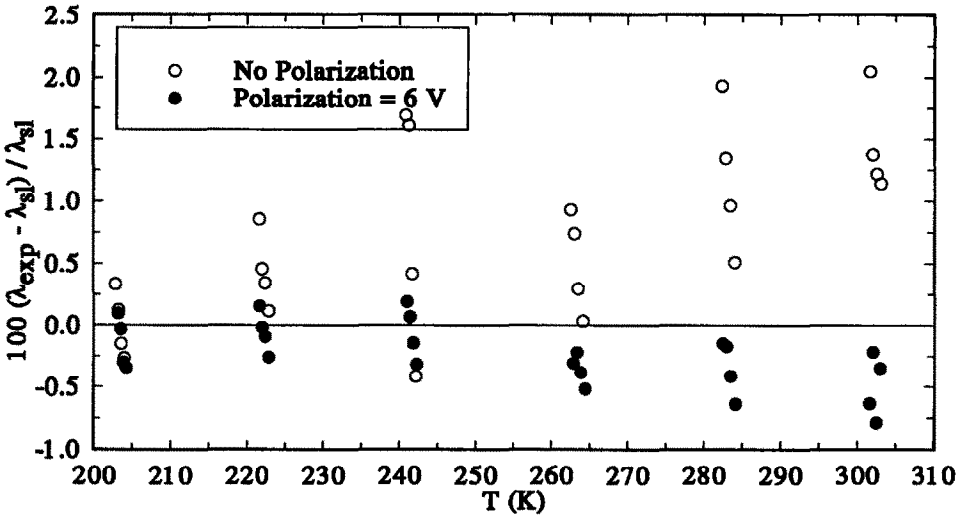

Fig. 4. Comparison between the unpolarized and polarized thermal conductivity data and the saturated liquid thermal conductivity fit of eqn. (2) for R134a.

nonpolarized measurements. There is a trend of increasing discrepancy between polarized and nonpolarized experiments as the temperature increases. This may be due to an increased mobility of ionic impurities in the electric field as the temperature is increased.

Comparison with other techniques

The thermal conductivity data for R142b from Table 3 are compared with the steady-state concentric cylinder data of Sousa et al. (1991) in Figure 5. The baseline for the comparison in Figure 5 is the surface correlation of Sousa et al. (1991). The uncertainty in the Sousa et al. (1991) data for R142b is ±2% while our uncertainty for a polarized measurement is about ±1%. Our polarized thermal conductivity data are higher than the Sousa et al. (1991) concentric-cylinder data by about 1% which is well within the mutual uncertainty of the two techniques. The unpolarized transient hot-wire data are systematically higher than the Sousa et al. (1991) data by up to 4% with a repeatability as a function of applied power of ±1%.
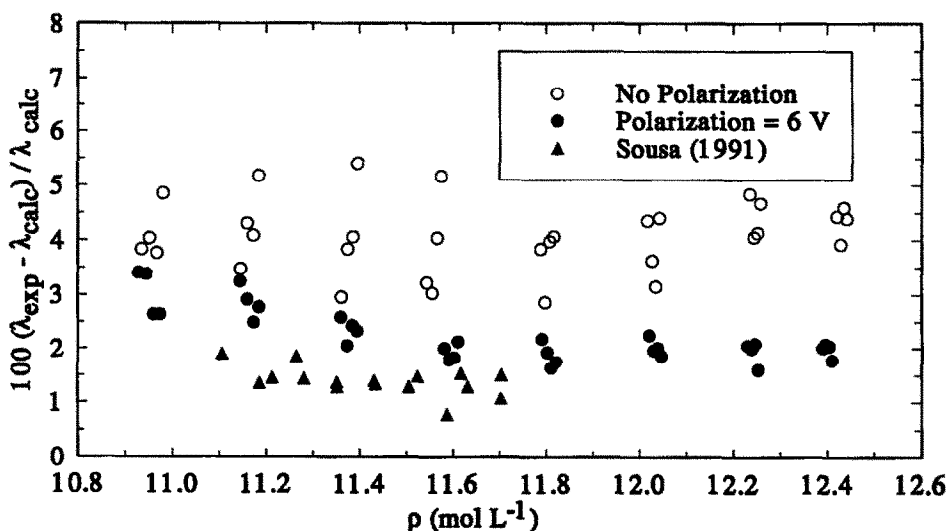

Fig. 5. Comparison of our nonpolarized and polarized thermal conductivity data with the steady state concentric cylinder data of Sousa et al. (1991) for R142b near 300 K. The comparisons are relative to the correlation of Sousa et al. (1991).

CONCLUSIONS

The increased uncertainty in transient hot-wire measurements made on polar fluids with bare wires is thought to be due to dilute ionic impurities which are soluble in the liquid phase. The presence of these impurities can be detected by measuring the electrical resistance or the electric potential between the hot wires and the cell walls. These ionic impurities lead to the formation of an electric double layer near the surface of the hot wires whose presence introduces a measurement error to the transient hot-wire technique. The magnitude of the measurement error increases with increasing temperature and decreasing temperature rise.

This uncertainty can be reduced by application of a fixed dc polarization voltage between the hot wires and the cell walls. Once a minimum threshold polarization voltage is reached, there is little effect as the polarization voltage is increased. Polarized transient hot-wire measurements on polar fluids have scatter which is comparable to transient hot-wire measurements on nonpolar fluids. Agreement between polarized transient hot-wire measurements and steady-state concentric-cylinder measurements for R142b is within 1%, while nonpolarized transient hot-wire measurements produce thermal conductivity data which are too large by up to 4%.

ACKNOWLEDGEMENTS

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