Measurement of Thermal Diffusivity at Low Temperature

Using an Optical Reflectivity Technique[1]

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ABSTRACT

An experimental arrangement has been developed for measuring the transient temperature responses and the thermal diffusivities of foil materials in range of 10 K to 300 K by using the optical reflectivity technique. The cryogenic system with optical windows is installed to provide a temperature of experiment from 10 to 300 K. The front surface of a foil specimen is heated by a pulsed Nd:YAG laser. *In situ* measurement of the reflectivity of a continuous wave He-Ne laser at the rear surface is conducted at the microsecond time scale. Using the temperature dependence of reflectivity, the transient temperature response is deduced. The thermal diffusivity is obtained by fitting Parker′s formulae to the experimental data of temperature rise. The stainless steel foils are chosen as samples and at the measuring temperatures are set in the region from 10 K to 300 K. The accuracy is examined by comparing the present results with the theoretical temperature responses and the thermal diffusivity data from the literatures. Good agreement is obtained.

KEY WORDS: optical reflectivity technique; transient temperature response; thermal diffusivity; low temperature; stainless steel foil.

1. INTRODUCTION

The extensive applications of cryogenic technologies to the technical and industrial areas, such as the microelectronic devices cooling and the cryogenic vacuum systems, need the thermophysical properties at low temperature for successful design of facilities and production of the materials. The transient temperature measurement technique at low temperature can provide not only a method for measuring thermal diffusivity [1] but also an opportunity for observing some new phenomena in heat conduction [2, 3]. At low-temperature region, however, since the conventional infrared radiation technique is no longer valid, a new measuring technique is needed to develop.

The change in temperature affects the complex refractive index [4], which in turn influences the optical reflectivity of material. Accordingly the time-resolved reflectivity measurement will result to reveal the transient temperature response. The use of optical reflectivity technique offers a possibility for measuring the high-speed temperature response in the temperature region exceeding the waveband limits of infrared detectors, for example the low temperature. There are two techniques, in general, for the time-resolved reflectivity measurements: the modulated heating-pulse probing technique [5-7] and non-modulated heating-continuous wave probing technique [8-11]. The first method can overcome the response limits of the photo-detectors and can measure small signals of temperature changes by using modulation technique, but cannot be used to measure a temperature profile with slow decreasing. The second method can be used for any shape of temperature profile, but has difficulty to measure a temperature change faster than the response of photo-electronic sensor. The pulse probing technique has been used successfully to measure the transient reflectivity changes caused by electron temperature changes in metals irradiated by ultra-short laser pulse [6, 11]. The continuous wave probing technique has been used for transient temperature measurement of semiconductors in the range higher than room temperature. For the measurement of the transient temperature response and the thermal diffusivity at low temperature by using this technique, however, the work is seldom found in the literature.

This work develops an experimental arrangement for measuring the transient temperature response in the temperature range of 10 K to 300 K by using the continuous wave probing technique. The front surface of a foil specimen is heated by a laser-pulse irradiation. *In situ* measurement of the reflectance signal of a continuous wave laser at the rear surface is conducted and used to deduce the temperature response at the microsecond time scale. The thermal diffusivity is obtained by using the measured temperature rise. The stainless steel foils are taken as samples. The accuracy of the method is examined by comparing the present results with the theoretical temperature responses and the thermal diffusivity data from the literatures.

2. EXPERIMENT

2.1. Principle of measurement

The transient temperature measurement by optical reflectivity technique is based on the variation of the materials complex refractive index [4], $N = n(T,\lambda) + ik(T,\lambda)$, with temperature. The complex refractive index is related to the reflectivity by the Fresnel formulae [12], which is given by

$$R(T,\lambda) = \frac{\left(n(T,\lambda)-1\right)^2 + k^2(T,\lambda)}{\left(n(T,\lambda)+1\right)^2 + k^2(T,\lambda)} \qquad (1)$$

at normal incidence of light, where λ is the wavelength of incident light and T is the temperature of surface. For an incident light with a fixed wavelength, the reflectivity changes simply with temperature. If the reflectivity-temperature relation is known, the time-resolved reflectivity measurement will result to reveal the transient temperature response. For a big temperature change, the temperature dependence of reflectivity of the sample is basically necessary for deducing the temperature from reflectivity. This is usually determined by the ellipsometric measurement [13] or quantum mechanical calculation with the band structure [14]. Some results in literatures show that, for metals [14], semiconductors [15], and superconductors [16], the reflectivity are linearly proportional to the temperatures approximately.

For a small temperature change and a fixed wavelength, the reflectivity change as a function of temperature change on the surface of the sample can be written in form of Taylor series expansion as

$$\begin{aligned}\Delta R(T,\lambda) &= R(T_0+\Delta T,\lambda) - R(T_0,\lambda) \\ &= R'(T_0,\lambda)\Delta T + \frac{1}{2}R''(T_0,\lambda)(\Delta T)^2 + \Lambda \approx R'(T_0,\lambda)\Delta T\end{aligned} \tag{2}$$

where T_0 is the initial temperature and ΔT is the temperature change. According to Eq. (2), when the *in situ* reflectivity measurement is used to obtain only the temperature change history without need to know the temperature values, the temperature-time curve can be directly deduced from the normalized reflectivity-time curve.

2.2. Experimental setup

The experimental setup for the *in situ* optical reflectivity measurement at low-temperature is shown in Fig. 1. The cryostat with optical windows is installed to provide an environment temperature of experiment from 10 K to 300 K. The temperature at the sample holder is set by a microprocessor-based digital temperature indicator/controller with a silicon diode sensor. A pulsed Nd: YAG laser with wavelength of 1064 nm is used as a heating source. The pulse duration of the laser beam is measured to be 17 ns, using a silicon PIN photodiode with rise and fall time of 0.2 ns and a digitizing oscilloscope with 500 MHz sampling speed. The output energy of single pulse is measured in the range of 250-300 mJ by a power meter. The laser beam spot with diameter of 8 mm can cover the surface of the sample through the front optical window on the cryostat. In this situation, the temperature rise in the sample is estimated below 5 K.

A continuous wave He-Ne laser is employed as a probing light source for the reflectance measurement. Because previous work [9] did not shown obvious superiority of p-polarized light comparing with the unpolarized one, the unpolarized laser is used here. The power of probing laser is 2 mW, which could be weak enough that the temperature rise caused by it can be ignored. The He-Ne laser beam is expanded to 1.8 mm by a beam expander and led to the center of the rear surface of the sample by a prism and the rear optical window on the cryostat. The angle of incidence is approximately 0 deg. The normal incidence and the expanding of probing laser beam can decrease the error caused by thermoelastic displacement of the sample. The reflected light is collected by a beam expander set in an opposite direction, passes through a narrow-band interference filter, and then goes into a fast silicon PIN photodiode. The filter can avoid to sensing the lights not from the probing laser, such as the heating laser, thermal radiation from sample, and other light sources. Since the change rate of the reflectivity on temperature is very small, the photo-electronic circuit is designed to output only the signals indicating the changes in reflectivity. The output signals are amplified by a low-noise preamplifier, with a gain of 1-10000 and frequency band of DC-1 MHz, and recorded on a SONY-Tektronix TDS520 two-channel digital storage oscilloscope with the highest sampling speed of 1 GS/s. A silicon PIN photodiode is put at the front of the optical window for the incidence of heating laser, which gives a trigger signal to determine the initial point of the reflectance response.

3. RESULTS AND DISCUSSIONS

3.1. Temperature responses

The SUS304 (Cr: 18-20%, Ni: 8-11%, Mn: <2%, Si: <1%, Fe: balance) stainless steel foil with thickness of 90 μm is taken as a sample. The front surface is heated by a Nd:YAG laser pulse, the transient optical reflectivity change at the rear surface is probed by a continuous wave He-Ne laser. According to the measuring principle (Eq. (2)), because the temperature rise is small, the normalized temperature response can be deduced from the normalized transient reflectivity change directly.

Figure 2 shows the normalized transient reflectivity changes, as the normalized temperature responses, measured at 10 K, 50 K, 150 K, 250 K, and 290 K. At low temperature, a bigger fluctuation noise can be observed in the response curves, which is mainly caused by the thermoelastic displacement but not electric noise. This has been confirmed by changing the holder and the thickness of the sample. With temperature decreasing down to low, the change rate of reflectivity may become small and the fixture of sample may become loose, all these can result in the increasing of the fluctuation noise. This noise has decreased by expanding the probing beam and improving the fixture of sample, but it is still the main error source in the measurements.

In the figure, the theoretical fitting curves, which are obtained by using Parker′s formulae of normalized temperature response [1]

$$\Delta V(t) = 1 + 2\sum_{n=1}^{\infty}(-1)^n \exp\left(-n^2\pi^2 at/L^2\right), \quad (3)$$

are also plotted for comparison. The good agreement between the experimental results and the theoretical calculations shows that the transient reflectivity changes reveal the temperature responses accurately. By comparing the temperature responses at different temperatures, it can be seen that, to decrease the initial temperature increases the rise speed of the temperature at rear surface. This is because of the increase of thermal diffusivity with decreasing temperature.

3.2. Thermal diffusivities

Utilizing Eq. (3), thermal diffusivities can be estimated from the measured temperature responses. Figure 3 shows the thermal diffusivities of SUS304 stainless steel foils in the temperature range from 10 K to 300 K. In the figure, each plotted result, which is an average value obtained by four times of measurements, has a relative deviation below 5%. According to the results, thermal diffusivity increases slightly with decreasing temperature from 300 K to 100 K. While in the low-temperature region (below 50 K), a steep increase in thermal diffusivity with decreasing temperature is observed.

Some literature results are plotted in the figure for comparison. Unfortunately only two reference data of thermal diffusivities, at 300 K [17] and 10 K [18], could be found for SUS304 stainless steel. The thermal diffusivities of SMR1460 stainless steel are also plotted as reference data [19]. The others values are obtained by using the thermal conductivities [18, 20], the specific heat [21, 22], and the densities of some similar kinds of stainless steels. Here, the densities at various temperatures are estimated from the values of the thermal expansion coefficients [18, 23]. Some details about the reference data can be found in Table I. As can be seen in the figure, the present results agree with most of the literature data accurately at all temperature regions, but are quite different from the results (3) and (4) at the low-temperature region.

4. CONCLUDING REMARKS

The transient temperature responses of SUS304 stainless steel foils under pulsed laser heating have been measured in the temperature range of 10 K to 300 K by using the *in situ* optical reflectivity technique. The thermal diffusivities are obtained by fitting Parker′s formulae with the measured temperature rise. Good agreement between the experimental results and the literature data shows the present method being available for measuring thermal diffusivity at low temperature. Extending the time scale of measurement from the present microsecond to nanosecond may provide an opportunity for observing some new phenomena in heat conduction.

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Table I. Reference Data Sources in Fig. 3.

	Thermal Conductivity	Specific Heat	Thermal Expansion Coefficient
(1)	Ref. [18], SUS304	Ref. [21], SUS305	Ref. [18], SUS304
(2)	Ref. [18], SUS304	Ref. [22]	Ref. [18], SUS304
(3)	Ref. [20], SRM735	Ref. [21], SUS305	Ref. [23], SUS304L
(4)	Ref. [20], SRM735	Ref. [22]	Ref. [23], SUS304L

FIGURE CAPTIONS

Fig. 1. Experimental setup for transient optical reflectivity measurement during pulse-laser heating at low temperature.

photo detector; Optical filter; Prism; He-Ne laser;

Cryostat; Sample; Temperature controller; Compressor;

Preamplifier; Oscilloscope; Nd: YAG laser; Beam expander.

Fig. 2. Normalized transient optical reflectivity changes, as normalized temperature responses, of 90 μm SUS304 stainless steel foils under laser-pulse heating at various temperatures.

Fig. 3. Thermal diffusivities of SUS304 stainless steel foils in the temperature range from 10 K to 290 K obtained from the transient reflectivity measurements of temperature responses.

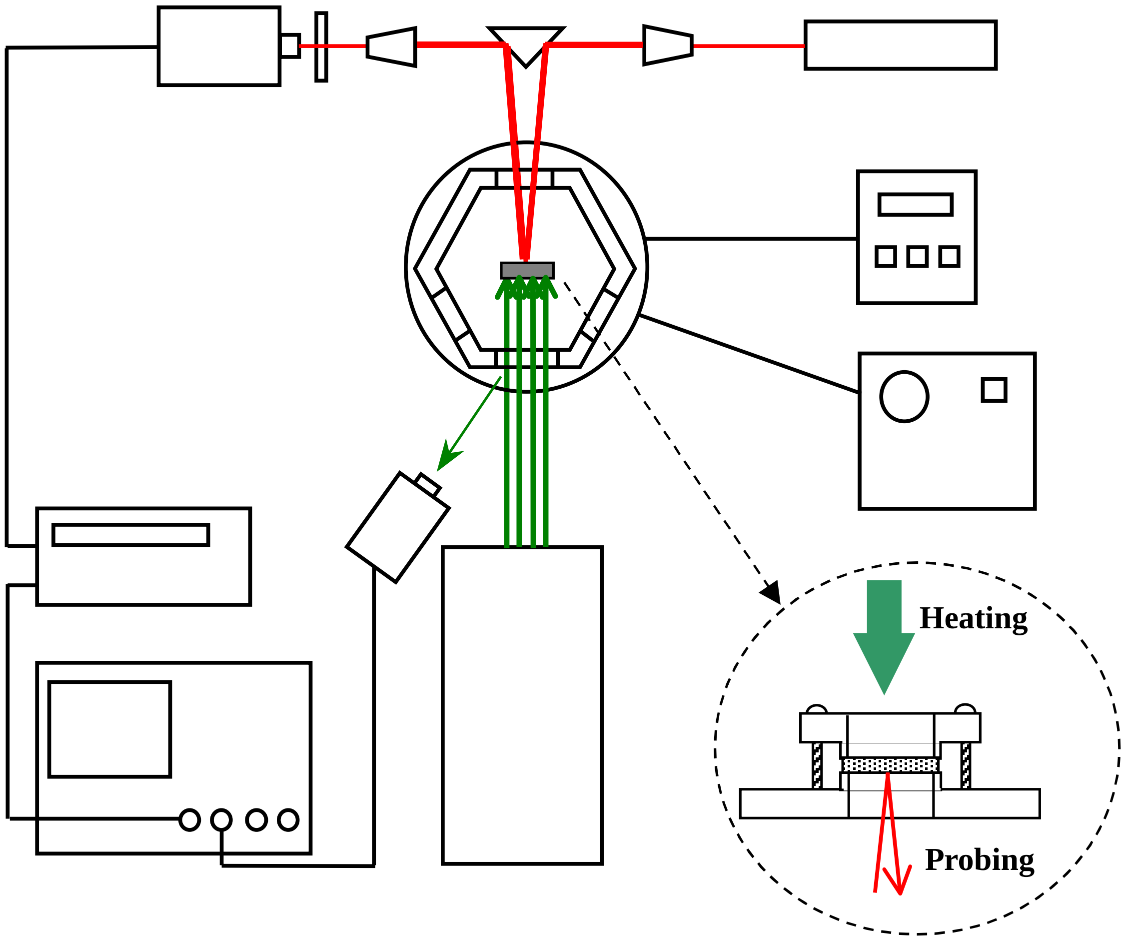


Fig. 1

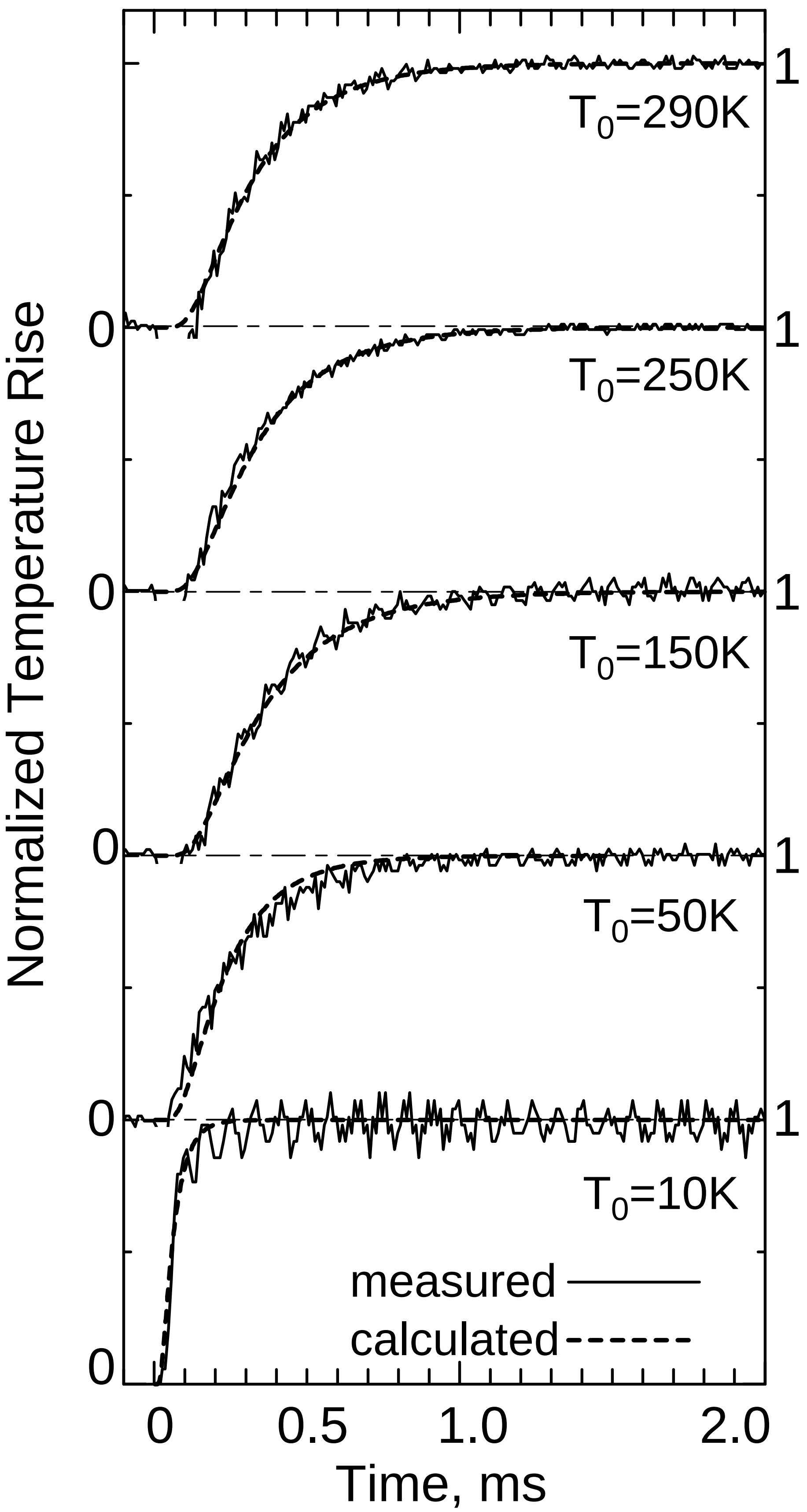
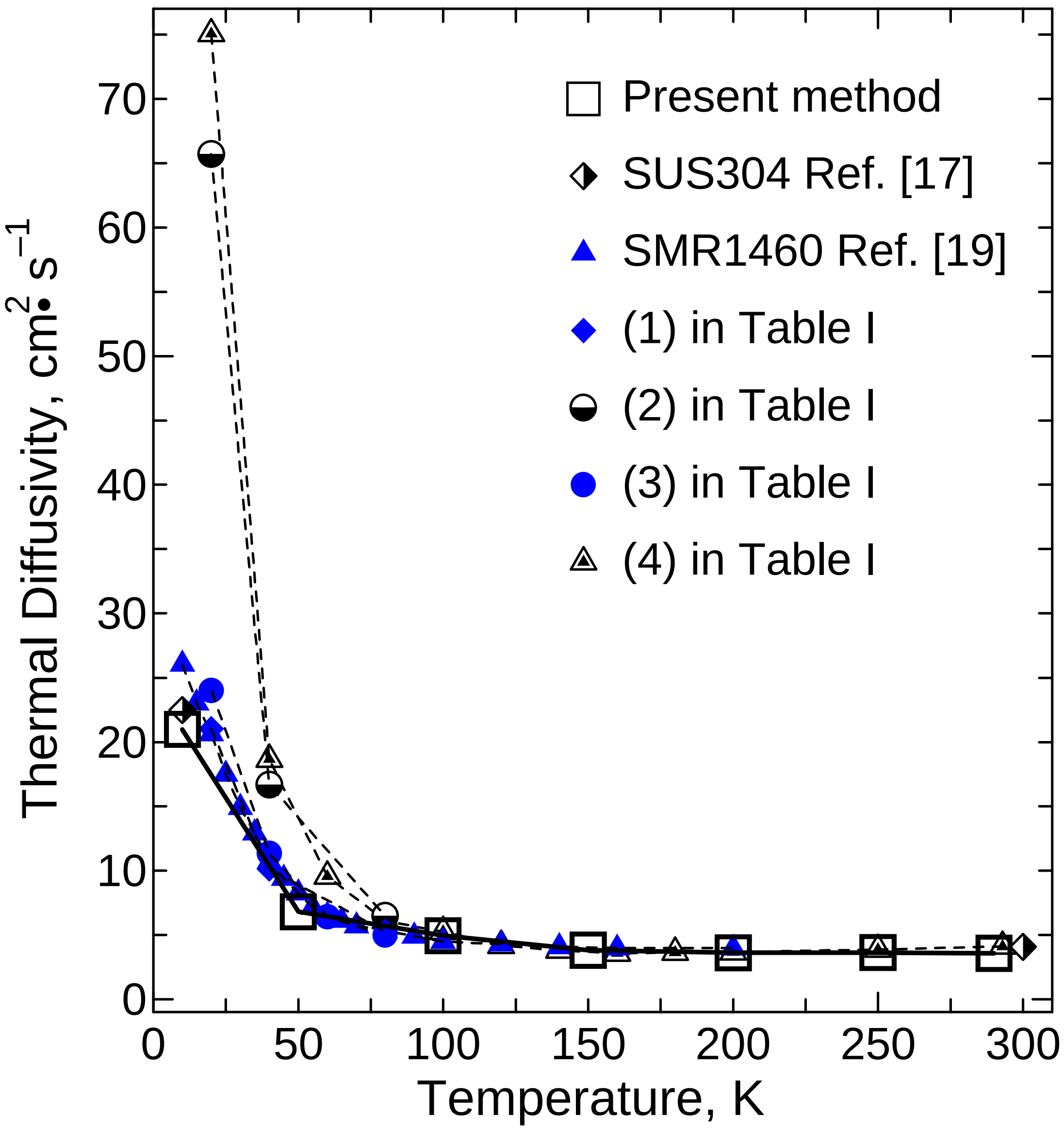

Normalized Temperature Rise
0
0
0
0
0
1
1
1
1
1
T_0=290K
T_0=250K
T_0=150K
T_0=50K
T_0=10K
measured
calculated
0
0.5
1.0
2.0
Time, ms

Fig.2



Present method
SUS304 Ref. [17]
SMR1460 Ref. [19]
(1) in Table I
(2) in Table I
(3) in Table I
(4) in Table I
Thermal Diffusivity, cm²• s⁻¹
Temperature, K
0
10
20
30
40
50
60
70
0
50
100
150
200
250
300

Fig. 3