PROGRESS REPORT

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OPTICAL STUDIES OF THE LITHIUM DEFECT COMPLEX IN IRRADIATED SILICON

Rensselaer Polytechnic Institute

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OPTICAL STUDIES OF THE LITHIUM DEFECT COMPLEX IN IRRADIATED SILICON

by

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Division of Nuclear Engineering and Science
Rensselaer Polytechnic Institute
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I. INTRODUCTION

The research work to be reported on here was started June 15, 1968 and is concerned with a study of the lithium defect complex produced in silicon upon irradiation by 1.5 MeV and 45-50 MeV electrons. Thus far the main probe used to detect the defects is the infrared spectroscopy of defect absorption bands that arise in the association of lithium atoms and vacancies and/or interstitials produced in displacement of silicon atoms by the radiation. In this six month period the irradiations were performed with the sample temperature kept $\lesssim 22^{o}C$ during bombardment. The irradiations utilizing 1.5 MeV electrons were performed at the General Electric Research and Development Center using the Cathode Ray Accelerator, while the 45-50 MeV electron bombardments were performed on the RPI microwave Linac. Subsequent to irradiation the infrared spectra (1 to 50 microns) were taken with the sample kept at $300^{o}K$ and at $\approx 80^{o}K$. Isochronal annealing experiments were performed using 15 minute pulses up to temperatures of $\sim 800^{o}K$.

The following personnel were actively engaged in the research program:

<u>Faculty</u>: Dr. John C. Corelli (half time summer, 3/8 time academic year).

<u>Research Technician</u>: Mr. James W. Westhead.

<u>Graduate Student</u>: Miss Rosa Chiang.

The following published reports of our NASA supported research work appeared in the literature during the past six months:

"Radiation-Induced Photoconductivity of Silicon" A. H. Kalma and J. C. Corelli, Radiation Effects in Semiconductors, edited by F. L. Vook, Plenum Press p. 153 (1968).

"Lithium-Associated Defect Complexes in Silicon" J. C. Corelli, J. W. Westhead and R. Chiang Bull. Am. Phys. Soc., Vol 13, p. 359 (1968).

Since the results of the work we are presenting here are part of a program still in progress we shall not attempt detailed discussions and conclusions of the findings. Rather, we shall give all of the results we have obtained and make brief comments where necessary. Future experiments that are planned and work currently in progress will be described briefly in this report.

II. EXPERIMENTAL METHODS

The silicon samples used in this study were of dimensions in the range 1 to 1½ mm thickness widths of 5 to 8 mm and about 15 mm long and were prepared from 2 mm thick slices cut from commercially available* silicon ingots. The lithium was doped** into the silicon using diffusion procedures reported by Pell.[1] Stated briefly the process consists of applying a mixture of lithium in mineral oil on the sample surface and performing a 1 to 2 hour anneal at $\sim 450^{\circ}C$ to $500^{\circ}C$. For samples of ~ 1.5 mm thickness a 1 to 2 hour heat treatment is sufficient to insure a homogeneous distribution of lithium throughout the bulk of

* Floating-zone and crucible grown silicon of resistivity $\lesssim 500$ ohm-cm were obtained from Texas Instruments Inc. Dallas, Texas or General Diode Corp., Framingham, Mass.; high purity floating-zone silicon of $\gtrsim 25,000$ ohm-cm was obtained from Wacker Chem. Corp., 202 East 44th St., New York, N. Y.

** We thank you Mr. Y. M. Liu and M. W. Wappaus of the NASA Goddard Space Flight Center for performing the diffusions for us.

the sample. Four point resistivity measurements on opposite sample faces are made to give a check of sample homogeneity which in most cases is better than $\pm$ 10% assuming the value of resistivity is a measure of the lithium concentration.

A listing of the samples studied is given in Table I. The table gives the initial resistivity of the silicon, the lithium concentration and resistivity after diffusion. All samples had n-type conduction after diffusion. The first four samples listed in Table I and sample W 42-2 were irradiated to 50 MeV electron fluences of 1.8×10^{18} e/cm^2, sample W 42-1 was irradiated by 1.5 MeV electrons to a fluence of 1.4×10^{18} e/cm^2. The samples Liu #1, P-1, and Liu #3 were irradiated to 47 MeV electron fluences of 2.3×10^{18} e/cm^2. In the 45-50 MeV bombardments the samples were kept cool during irradiation ($< 20^oC$) by immersing them in a flow of ice cold water. The 1.5 MeV bombardment was made with the sample in contact with a thin walled aluminum box filled with ice water.

The infrared spectra were measured on samples kept at $\approx 80^oK$ in a glass ~~drawer~~ dewar having CsI windows, with room temperature measurements of the spectrum also taken routinely. The infrared spectrometers used were Perkin Elmer models 621 (2.5 to 50 microns) and 21 (1 to 15 microns). The annealing experiments were carried out in air in a temperature controlled furnace, and the samples were thoroughly cleaned and lapped after each heat treatment to eliminate the effects of oxide impurities at the surface.

TABLE I

PROPERTIES OF SILICON SAMPLES STUDIED

Sample Designation	ρ_o (ohm-cm) initial	lithium conc. cm^{-3}	ρ (ohm-cm) after lithium diffusion
Wacker #1 Li	28,500 FZ	2×10^{16}	0.27
Wacker #2 Li	28,500 FZ	8×10^{17}	0.028
Wacker #3 Li	28,500 FZ	1.2×10^{17}	0.08
Wacker #1 No. Li	28,500 FZ	NONE	28,500
W42-1	500* FZ	2.5×10^{17}	0.05
W42-2	500* FZ	2.5×10^{17}	0.05
Liu #1	20* FZ	2×10^{19}	0.003
P-1	200* CG	2×10^{16}	0.3
Liu #3	15** CG	1.5×10^{18}	0.02

* Phosphorus doped
** Boron doped
FZ floating zone refined
CG crucible grown

III. Results

The results to be given here are primarily concerned with defects that have long time stability ($\sim$1-3 days). Although as we shall see the radiation-produced defects appear to be complexes in which one of the constituents is a Li ion, none of the defects we are concerned with exhibits short term transitory annealing (say of order minutes or hours after ceasing the bombardment). Other researchers[2,3,4] have found this short term annealing in irradiated silicon solar cells where the diffusion length and carrier recombination times serve as detectors of the damage. In our case we observe the radiation-produced infrared active defect absorption bands a long time after irradiation, and the damage we are observing is stable up to $\sim 35^oC$. This main distinction in the stability of defects must be borne in mind when examining our results.

A. Long Wavelength (> 6 microns) Absorption in Li-diffused Si.

In Fig. 1 are shown the spectra in the 800 to 200 cm^{-1} (12.5 to 50 micron) wavelength region for a silicon sample W42-1 containing $\sim 10^{17}$ Li/cm^3 (See Table I). Each spectrum was measured after a fifteen minute heat treatment at the indicated temperature. Although not shown the spectra for annealing temperatures lower than 200^oC were also very similar to the 200^oC spectrum shown in Fig. 1 and are not shown. Note the evolution of a broad absorption band after the 400^oC anneal and its disappearance subsequent to higher temperature treatment.

This broad absorption band has not been exhibited by any of the other samples studied and would require further study before definitive statements and conclusions can be drawn about the broad band (250 to 400 cm^{-1}).

The spectra after 47 MeV electron irradiation of a Li-doped silicon sample Wacker #2 to a fluence of 1.8 x 10^{18} e/cm^2 at which 15 minute anneals occured is shown on each spectrum in Fig 2. The predominant absorption for frequencies below 600 cm^{-1} occurs after heat treatment to $\sim 100^{o}C$. It must also be emphasized that the sample exhibits a large absorption in the 200 to 400 cm^{-1} region even before heat treatment experiments. This absorption may be indicative of some free carriers which were not trapped by defects, which is reasonable if one compares the fluence to the initial lithium concentration. Other work[5,6] suggests carrier removal rates of ~ 1 to 5 cm^{-1} are to be expected in our case.

The long wavelength absorption exhibited by a crucible grown silicon sample Liu #3 (diffused with lithium) after 15 minute anneals at each of the indicated temperatures is shown in Fig. 3. The sample, Liu #3, was irradiated to a 47 MeV electron fluence of 1.8 x 10^{18} e/cm^2. The results given in Figs. 1 to 3 are for samples prepared from floating zone refined and from crucible grown silicon in which one very important difference is the relatively large oxygen concentration is crucible grown $\gtrsim 10^{17}$ O/cm^3 as compared with floating zone silicon $\lesssim 5 \times 10^{16}$ O/cm^3. The sharp bands in the 800 to 1000 cm^{-1} wavelength region are well known[7,8,9] oxygen-associated defect bands, and in particular the sharp band at $\approx$ 832 cm^{-1} the so-called A-center consists of a substitutional oxygen plus vacancy complex.[10] However, there are some striking differences in the results shown in Fig. 3 relative to what has been observed in previous studies on oxygen-defect complexes,[7,8,9] and that is the diappearance of the normal interstitial oxygen band at $\approx$ 1125 cm^{-1} and the oxygen-associated defect bands in the $\sim$800 to 1000 cm^{-1} wavelength region after 15 minute heat treatments to 270°C. The disappearance of these bands may be due to the release of free carriers upon annealing and the absorption accompanying free carriers. Apart from the oxygen associated defect bands, sample W42-2 a floating zone* lithium diffused sample irradiated by 48 MeV electrons to a fluence of $\sim 10^{18}$ e/cm^2 exhibits the same spectra and behavior as found for sample Liu #3 which is given in Fig. 3.

A more detailed presentation of the effects of heat treatment on the oxygen-associated defect bands for sample Liu #3 is given in Fig. 4 where we have plotted the transmission vs frequency in the 700 to 1200 cm^{-1} range. For purposes of comparison in Fig 5 we have plotted some similar annealing results for a crucible grown sample of silicon doped with bismuth to a resistivity of 0.01 ohm-cm. This Bi-doped silicon sample was irradiated by $\sim$48 MeV electrons at 300°K to a total

*The oxygen associated defect absorption bands are never observed in floating zone material due to the low initial oxygen concentration.

fluence of 2×10^{18} e/cm^2. It is interesting to note that for both cases the oxygen associated A-center band (826 cm^{-1}) disappears upon heat treatment to temperatures well below the 350°C[7,8,9] generally found in crucible grown silicon containing lower concentrations of donor and acceptor impurities i.e., having resistivity $\gtrsim$1 ohm-cm.

During the course of our annealing experiments on float-zone refined silicon we detected a new defect absorption band at 1122 cm^{-1} which first begins to appear after an anneal to $\sim$300°C. Another defect band at 1092 cm^{-1} of markedly less absorption also appears upon heat treatment to 330°C. The detailed results showing the evolution and disappearance of the 1122 cm^{-1} and 1092 cm^{-1} bands are given in Fig. 6 where we have plotted the transmission vs wavelength (1000 to 1300 cm^{-1} region) after 15 minute anneals at each of the temperatures shown. The temperatures shown for sample Wacker #1 (No Li) apply also to samples Wacker #1 and Wacker #3 in Fig. 6. By referring to Table I and Fig. 6 it can be seen that the presence of lithium impurity atoms has a pronounced effect on the 1122 cm^{-1} and the 1092 cm^{-1} bands. In fact for the most heavily doped Li-diffused silicon sample, as for sample Wacker #2, the bands at 1122 cm^{-1} and 1092 cm^{-1} are not observed. We have not observed such strong impurity effects on the defect absorption band in any of our previous work,[8,11] nor have other researchers reported such striking effects. Once again we have clear direct proof of the extremely important role that is assumed by impurities in their interaction with radiation produced defects.

B. Short Wavelength ($<$ 3 microns) Absorption in Li-Diffused Si

Careful investigation of the infrared spectrum from 1.25 to 2.5 microns revealed two new radiation-induced defect absorption bands. The new bands are located at 1.45 and 1.67 microns, as shown in Fig. 7. The spectra presented in Fig. 7 were all measured with the sample temperature kept at 300°K. The annealing temperature (15 minute temperature pulses) is shown on each spectrum. It is clearly evident from the results given in Fig. 7 that the presence of lithium impurity atoms strongly affects the defect absorption bands in that the 1.85 micron band which we have[11] identified as arising from the divacancy defect in silicon is not observable in the high concentration lithium diffused sample of 0.03 ohm-cm Wacker #2. Whereas, for the sample containing no lithium 25,000 ohm-cm Wacker #1 (No Li), only the divacancy[11] defect band at 1.85 microns is observed, and of course the so called "near edge absorption" can be seen easily in the 1.25 to 1.55 micron range. We[11] have associated the "near edge absorption" with disorder in the lattice structure of silicon. Such near edge absorption which generally extends from $\sim$1.2 to $\sim$4 microns has also been observed[12,13] in reactor neutron-irradiated silicon. In recent photoconductivity studies on 45 MeV electron-irradiated silicon we[14] have observed dominant defect photoconductivity by an "energy band" rather than by single well defined energies as found in 1.5 MeV electron irradiations of silicon. We have suggested that the energy band is due to disorder regions in the lattice which destroy a good deal of the periodicity of the lattice, thus having the effect of destroying the sharp division between allowed and forbidden energies at the band edges.

The infrared spectra in the 1 to 2.5 micron range of two crucible grown Li-diffused silicon samples is shown in Fig. 8. Both samples were exposed to a 47 MeV electron fluence of 2.3 x 10^{18} e/cm^2. The spectra shown in Fig. 8 were measured at 300°K and after 15 minute anneals at each of the indicated temperatures. Here again we can see that the lithium concentration has a pronounced effect on the 1.48 micron defect absorption band. Note that for the case of the lower lithium concentration sample P-1 we can clearly see the 1.85μ divacancy defect absorption band, whereas for the case of the higher lithium concentration sample Liu #3 no divacancies are formed as evident by the lack of the divacancy absorption band at 1.8 microns.

It is extremely important to recognize that the spectra we have presented in Figs. 7 and 8 point to a need for higher energy resolution in this very crucial wavelength range. At the present time we are building on input and an output optics system to be used in conjunction with a high resolution grating monochromator* acquired within the past two years.

*The monochromator is a Spex model 1500 Evacuable Monochromator, Spex Industries, Inc. 3880 Park Ave., Metuchen, New Jersey.

IV. DISCUSSION OF RESULTS

The most striking results of this work thus far point to the fact that for high lithium doping concentrations, the lithium atoms apparently combine with other defects such as the divacancy, the A-center etc. thereby producing either a new complex of more constituents or the Li atoms quickly combine with single vacancies (or multiple vacancies) during the time of production of vacancies etc. thereby immobilizing the vacancies and prohibiting them from exhibiting additional further motion in the lattice. A clear example of the impurity interactions with the defect bands is given in Fig. 4 where the oxygen associated A-center band recovers after a 15 minute anneal at 200°C rather than 350°C as found[7,8,9] in silicon of $\lesssim 10^{16}\ \frac{\text{impurity atoms}}{\text{cm}^3}$. It is significant to note that the band at 770 cm^{-1} in Fig. 4 must arise from a defect complex having at least one oxygen plus one lithium atom as constituents. Of course the presence of at least one vacancy in the complex is possible. The requirement of the presence of oxygen atoms in addition to lithium atoms for the appearance of bands at 770 cm^{-1}, 855 cm^{-1}, 930 cm^{-1} and 1006 cm^{-1} can be seen if one compares the results of Fig. 4 for crucible grown silicon to the spectra obtained from floating zone silicon (Li-doped) and given in Figs. 1 and 2. The band at 1006 cm^{-1} has been studied in detail by Pell[15] and by Chrenko, McDonald, and Pell.[16] These workers concluded that the 1006 cm^{-1} band is a vibrational band arising from a lithium plus oxygen complex OLi^{+}, in which the oxygen interstitial band at 1106 cm^{-1} is displaced to 1006 cm^{-1} by the presence of Li (both Li-6 and Li-7). Our results in Figs. 2 and 4 agree with Pell's findings in which Li and O concentrations $\gtrsim 10^{17}$ give rise to the 1006 cm^{-1} consisting of OLi^{+}

At this point we can only give speculative arguments relative to the complexes giving rise to the 1.48 and the 1.7 micron defect absorption bands. The bands at 1.48 and 1.7 microns might be due to complexes consisting of one and two Li^{+} ions respectively which are trapped by a divacancy since the 1.85 micron band decreases with the appearance of the 1.48 band and the 1.85 disappears completely when the 1.7 micron band first appears. In Fig. 7 we can observe that there is a distinct dependence of the annealing of the 1.48μ band on the lithium concentration and in fact appears to be unstable for the sample containing the highest lithium concentration, namely Wacker #2 0.03 ohm-cm insofar as the recovery occurs at a lower temperature $\sim 150^{\circ}C$ as opposed to $\sim 330^{\circ}C$ for the sample containing the lowest lithium concentration Wacker #1 0.27 ohm-cm. These temperature effects are also observed for the 1.7 micron band and may very well be due to the effects of free carrier absorption masking the absorption in the 1.48 and 1.7 micron bands.

We conclude that the new radiation-induced bands in high oxygen $\gtrsim 10^{17}$ cm^{-3} high lithium $\gtrsim 10^{17}$ cm^{-3} containing silicon located at 1006 cm^{-1}, 930 cm^{-1}, 855 cm^{-1} and 770 cm^{-1} must arise from defects that consist of both lithium plus oxygen atoms in addition to vacancies and/or interstitials since sample P-1 (see Table I) which contains $\gtrsim 10^{17}$ oxygen/cm^3 but a relatively low concentration of lithium $\sim 2 \times 10^{16}$ Li/cm3 exhibited none of the just mentioned bands. In fact the sample** P-1 only exhibited the normal A-center band at $\approx$ 830 cm^{-1} and its annealing at 350°C (15 minutes) with no other band appearing in the wavelength range 800 to 1100 cm^{-1}.

We wish to conclude this discussion with a statement on the new radiation-induced bands at 1122 cm^{-1} and at 1092 cm^{-1} which appear in float-zone-refined silicon after heat treatment to $\sim$ 300°C (see Fig. 6). The bands at 1122 cm^{-1} and 1092 cm^{-1} have not been observed previously and appear to be impurity independent and of course are not observable in silicon containing $\gtrsim 10^{17}$ $\frac{\text{oxygen}}{\text{cm}^3}$ since the interstitial oxygen band $\approx$ 1130 cm^{-1} masks out the 1122 cm^{-1} and 1092 cm^{-1} bands. It is tempting to ascribe the 1122 and 1092 cm^{-1} bands to a defect of higher order than the divacancy which anneals at exactly the temperature 300°C, at which the 1122 cm^{-1} and 1092 cm^{-1} bands appear. Mindful we are not on a firm base we propose that the bands in question may consist of a double divacancy or a trivacancy. However, much additional research work is needed to make a meaningful identification, and such work is now in the planning stages.

**The anneal results for the sample P-1 in the 800 to 1100 cm^{-1} range are not given here as they are exactly what has been been observed[7,8,9] in normal crucible grown silicon (oxygen $\gtrsim 10^{17}$ cm^{-3}).

V. EXPERIMENTS PLANNED AND IN PROGRESS

1. To study the effects of other impurities such as P, As, Bi and Sb on the divacancy defect band in the 1 to 4.5 micron range we shall irradiate some relatively impure silicon ~ 0.01 ohm-cm to fluences of 48 MeV electrons of $\sim 2 \times 10^{18}$ cm^{-2}. The radiation will be performed at 300°K and we shall only be concerned with defects and complexes stable at 300°K. We expect to get answers to the question why is Li as an impurity atom different in its interaction with radiation-produced defects in silicon from other donor impurities such as P, As, Sb or Bi.

2. We shall put a major effort during the next six month period on attempting to study the symmetry properties of the 1.48 and 1.7 micron Li-associated defect complixes in irradiated float-zone silicon. For these experiments we shall dope with lithium to concentrations $\sim 5 \times 10^{17}$ cm^{-3}. Samples will oriented in the [011], [001] and [111] directions and we shall search for stress induced dicroism in the infrared absorption bands utilizing the technique we used[11] successfully to study the 1.8, 3.3 and 3.9μ divacancy bands in silicon. Our first experiments will involve the application of stress at high temperature $\gtrsim 300$°K.

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FIGURE CAPTIONS

Figure 1 Infrared spectra (200 to 800 cm^{-1} range) of a Li-doped silicon sample W42-1 irradiated by 1.5 MeV electrons to a fluence of 1.4×10^{18} e/cm^2 after 15 minute anneals at each of the temperatures shown on the figure.

Figure 2 Infrared spectra (200 to 1200 cm^{-1}) of a Li-doped silicon sample Wacker #2 irradiated by 47 MeV electrons to a fluence of 1.8×10^{18} e/cm^2 after 15 minute anneals at each of the temperatures shown on the figure.

Figure 3 Infrared spectra (200 to 1600 cm^{-1}) of a Li-doped silicon sample Liu #3 irradiated by $\sim$47 MeV electrons to a fluence of $\sim 10^{18}$ e/cm^2 after 15 minute anneals at each of the temperatures shown on the figure.

Figure 4 Infrared spectra (700 to 1200 cm^{-1}) of a Li-doped silicon sample Liu #3 irradiated by $\sim$47 MeV electrons to a fluence of $\sim 10^{18}$ e/cm^2 after 15 minute anneals at each of the temperatures shown on the figure.

Figure 5 Infrared spectra of a crucible grown 0.01 ohm-cm Bi-doped silicon sample after 48 MeV electron irradiation to $\sim 10^{18}$ e/cm^2 and annealing for 15 minutes at each of the temperatures indicated.

Figure 6 Infrared spectra 1000 cm^{-1} to 1300 cm^{-1} for floating-zone-refined silicon samples containing various lithium concentrations. The spectra are shown after 15 minute anneals at each of the temperatures indicated on the figure.

Figure 7 Infrared spectra 1 to 2.5 microns for high purity floating-zone silicon samples containing various concentrations of lithium. Also shown are spectra for one high purity silicon sample. The samples were all irradiated to a 47 MeV electron fluence of 1.8×10^{18} e/cm^2, and after irradiation were subjected to 15 minute anneals at each of the temperatures indicated.

Figure 8 Infrared spectra for two Li-diffused crucible grown silicon samples after 47 MeV electron bombardment to a fluence of 2.3×10^{18} e/cm^2. The samples were given fifteen minute anneals at each of the temperatures indicated on the figure.

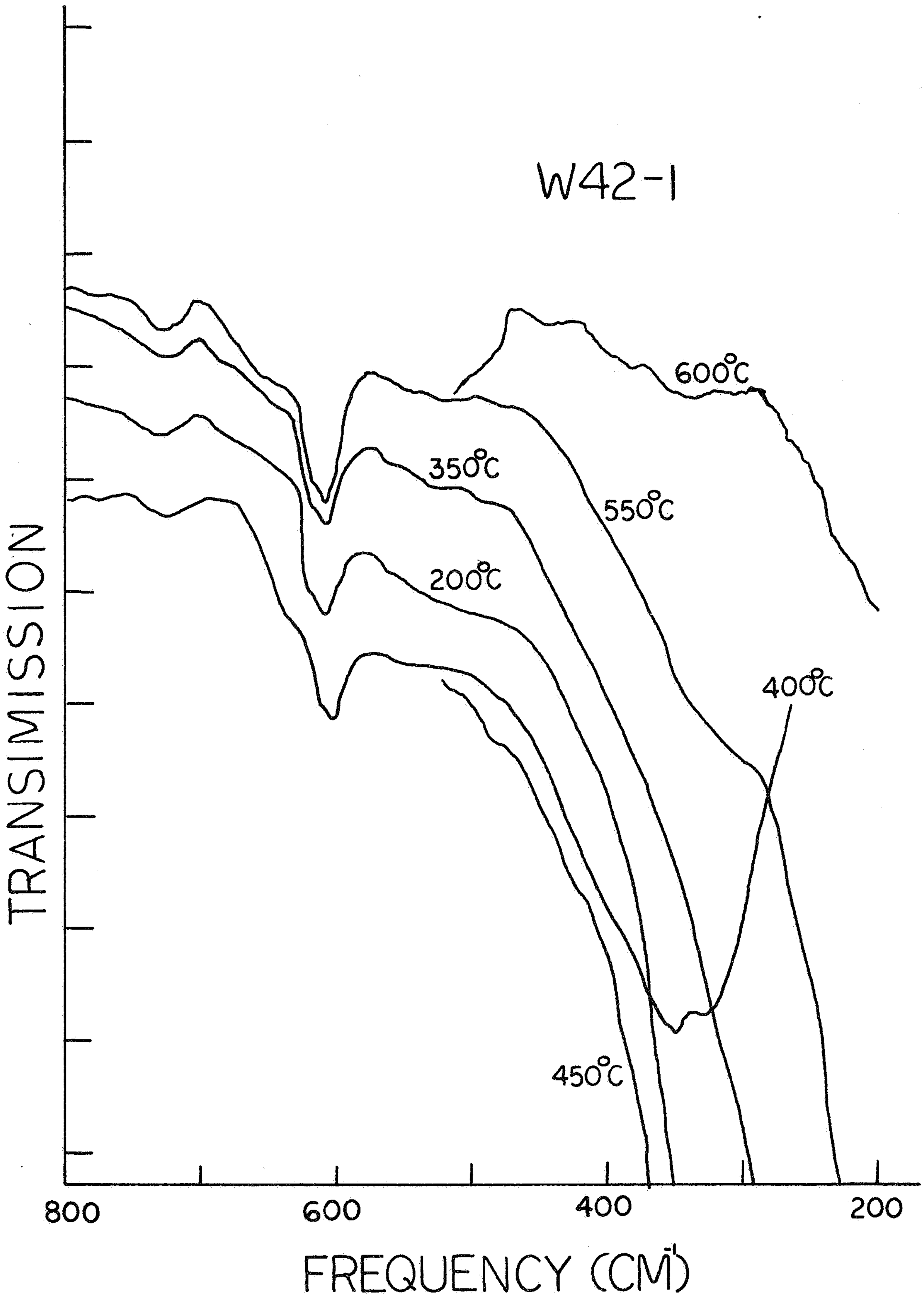
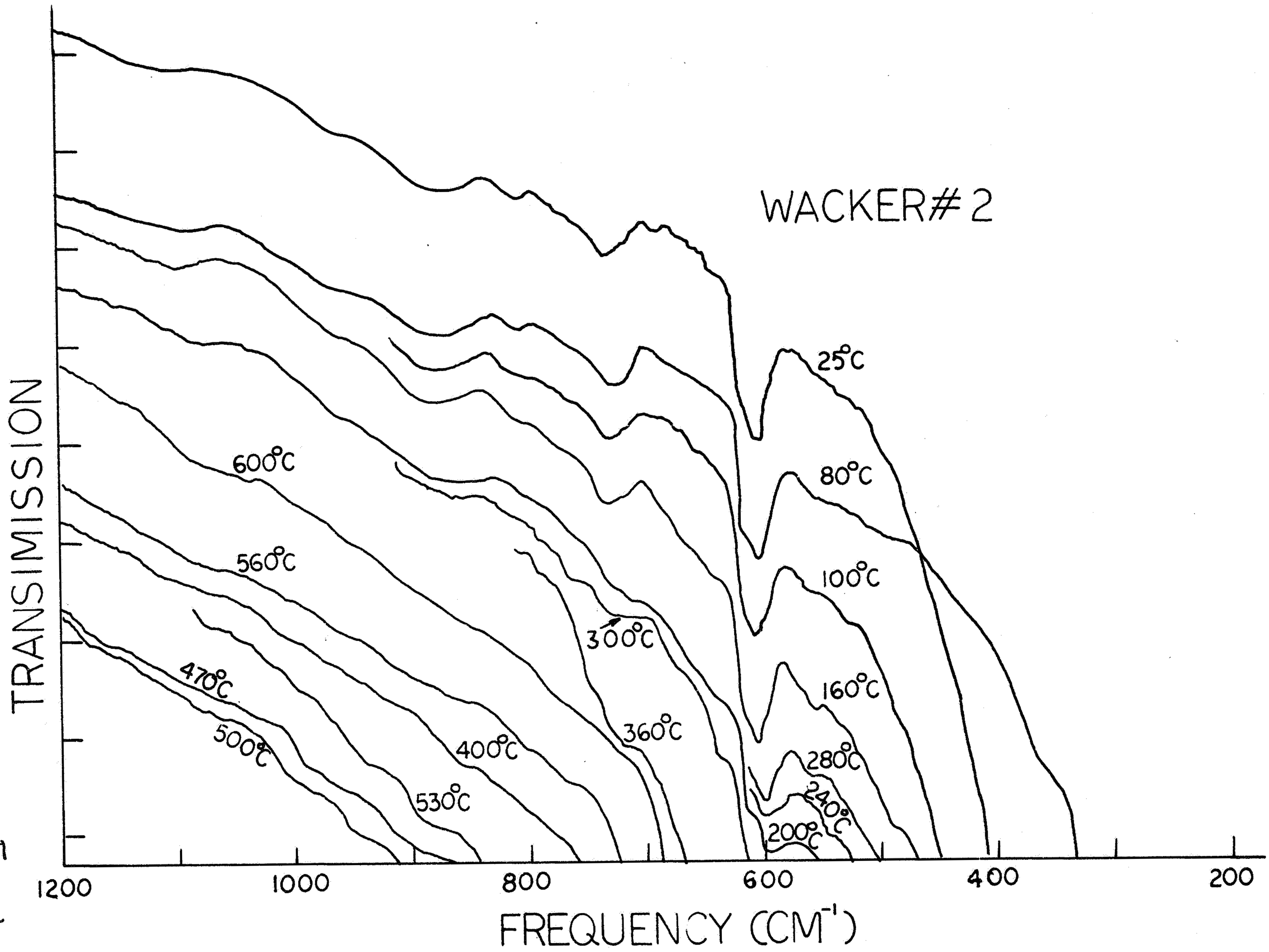

W42-1
TRANSIMISSION
FREQUENCY (CM⁻¹)
800
600
400
200
600°C
550°C
350°C
200°C
400°C
450°C

Fig. 1.)


WACKER# 2
TRANSIMISSION
FREQUENCY (CM^{-1})
1200
1000
800
600
400
200
25°C
80°C
100°C
160°C
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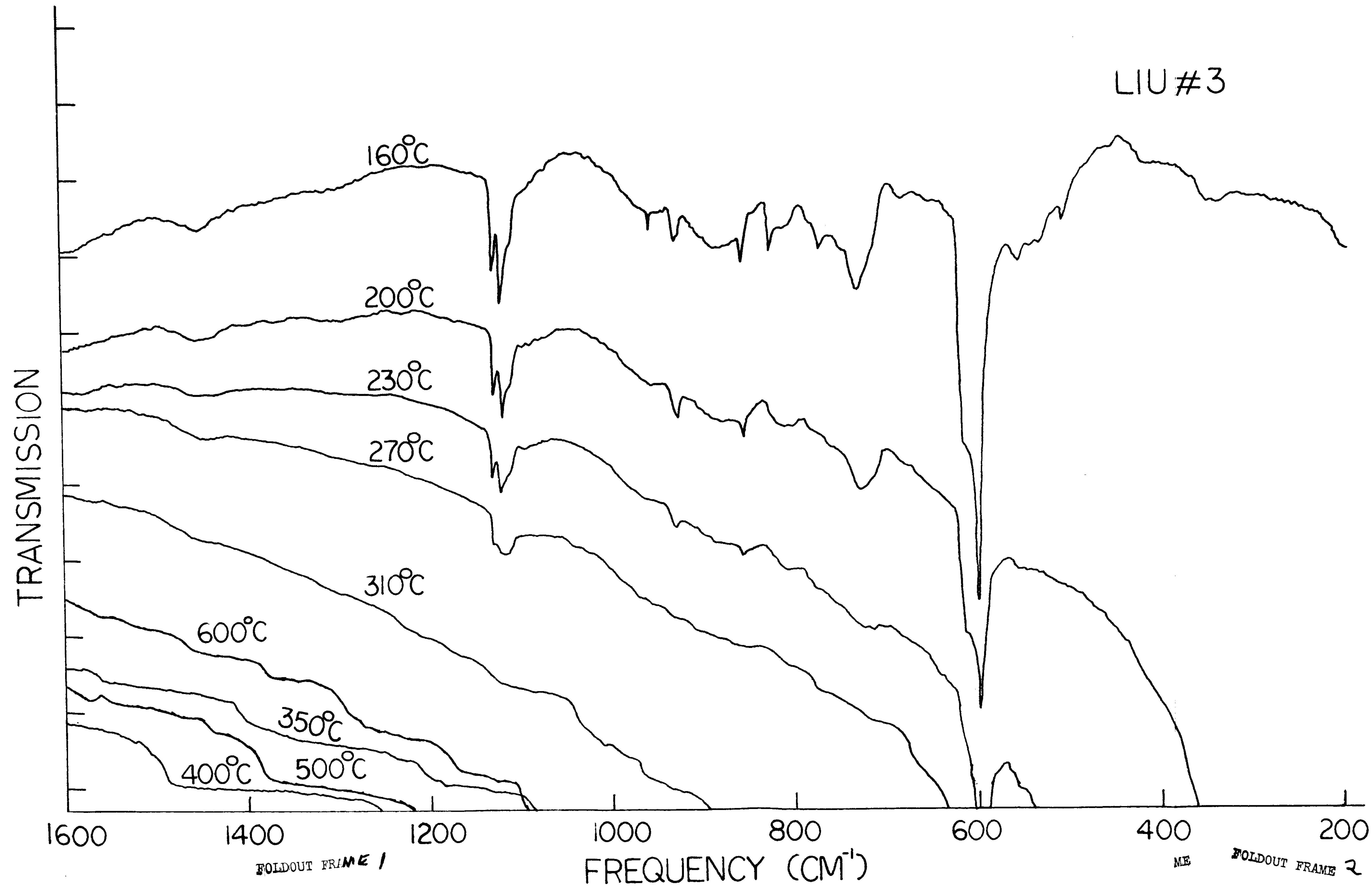
560°C
470°C
500°C

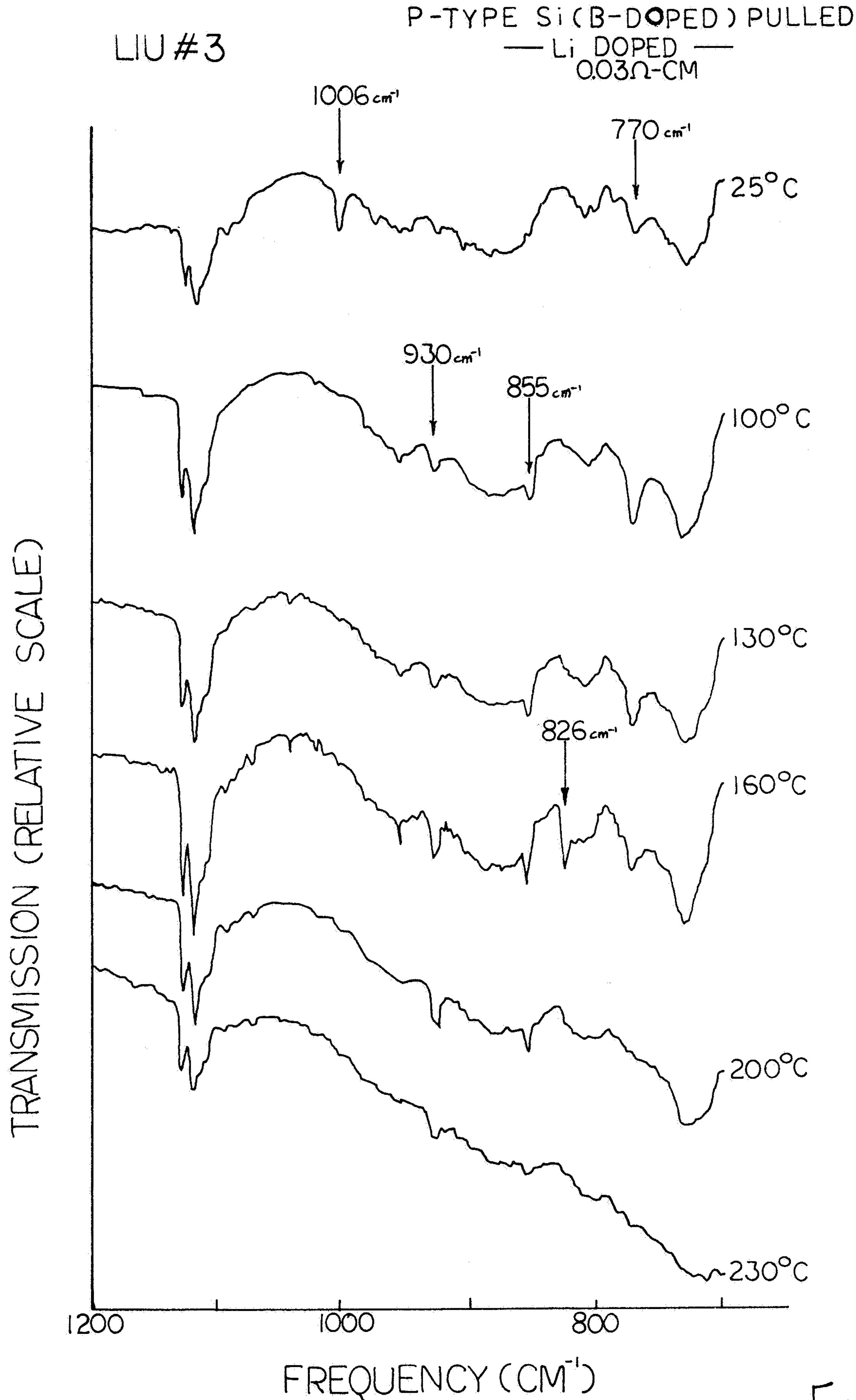
Fig 2.)



FOLDOUT FRAME 1

FOLDOUT FRAME 2

Fig 3


LIU #3
P-TYPE Si (B-DOPED) PULLED
— Li DOPED —
0.03Ω-CM
1006 cm⁻¹
770 cm⁻¹
25°C
930 cm⁻¹
855 cm⁻¹
100°C
130°C
826 cm⁻¹
160°C
200°C
230°C
TRANSMISSION (RELATIVE SCALE)
1200
1000
800
FREQUENCY (CM⁻¹)
Fig. 4)

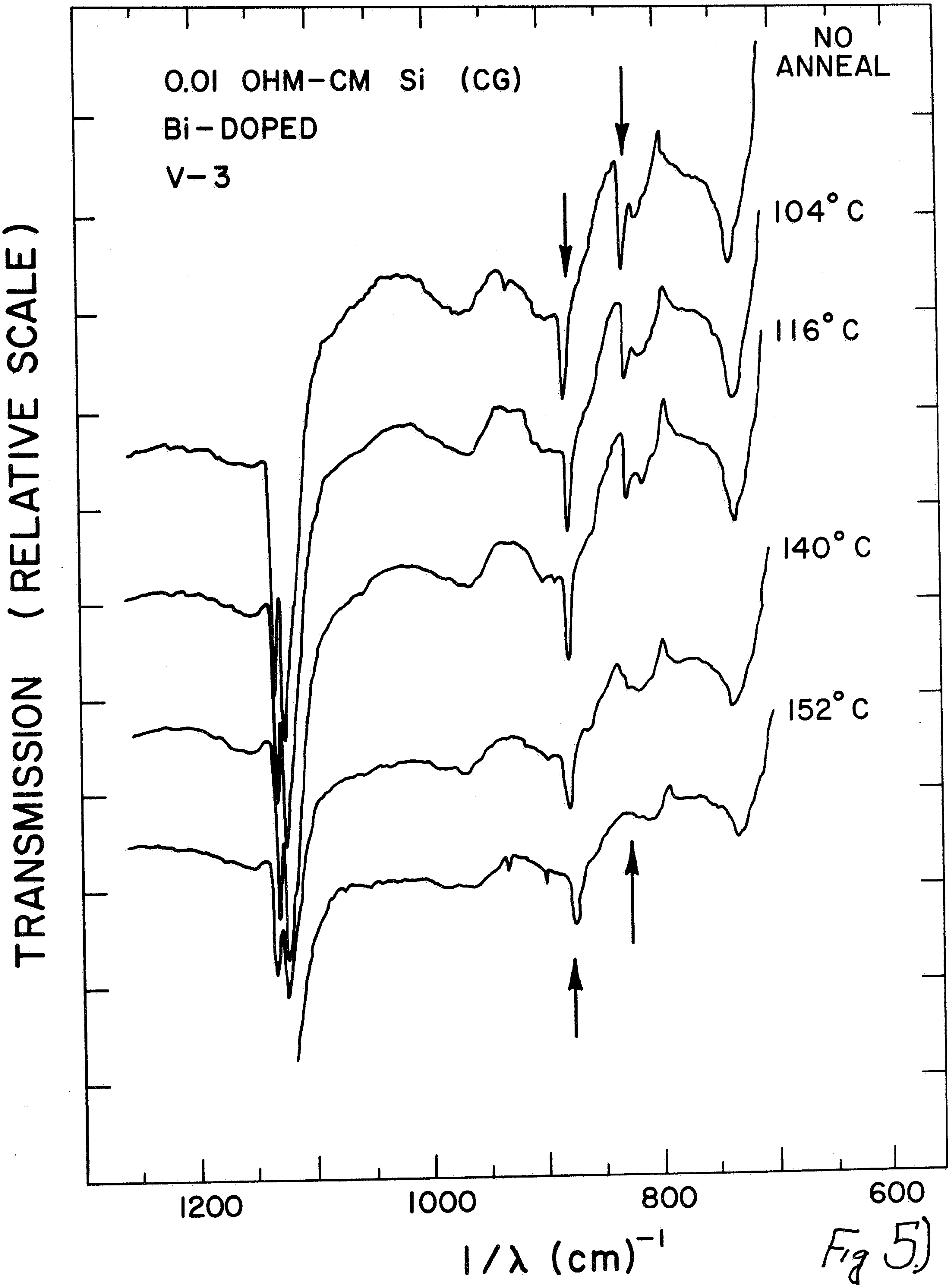

0.01 OHM-CM Si (CG)
Bi-DOPED
V-3
NO ANNEAL
104° C
116° C
140° C
152° C
TRANSMISSION (RELATIVE SCALE)
1200
1000
800
600
1/λ (cm)^-1

Fig 5.

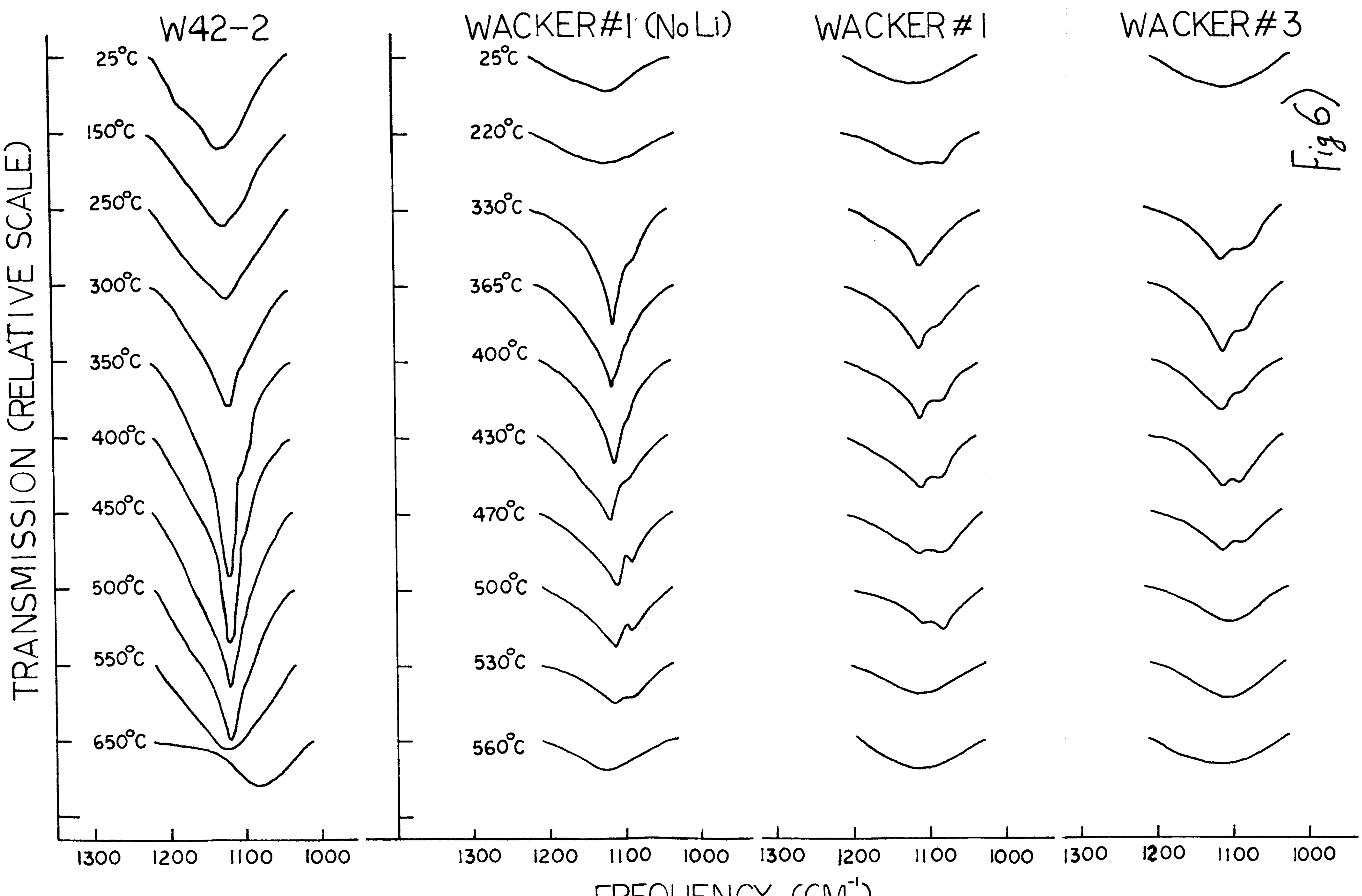
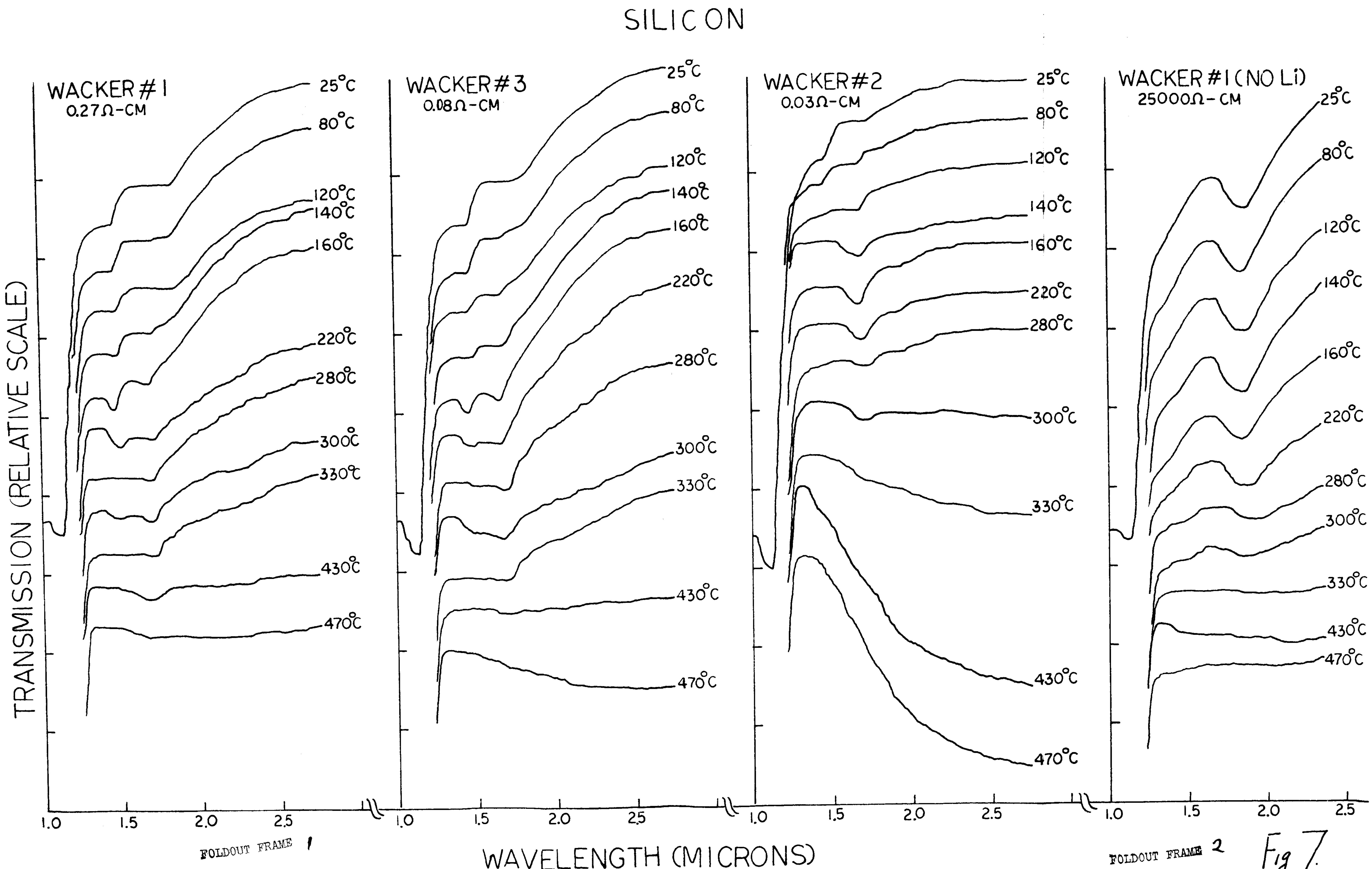


Fig 6)

FOLDOUT FRAME 1

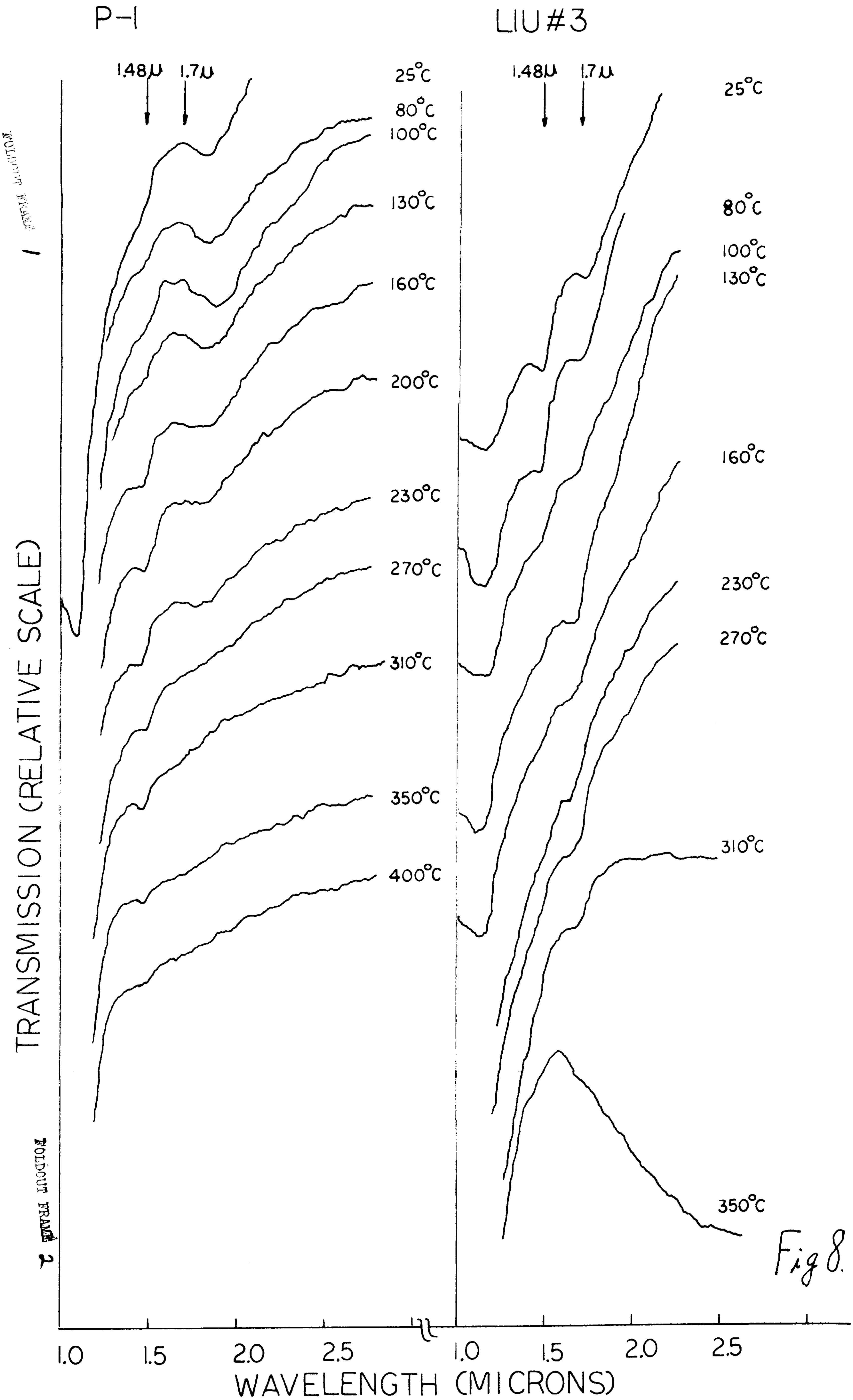
FOLDOUT FRAME 2



FOLDOUT FRAME 1

FOLDOUT FRAME 2

Fig 7.



P-1
LIU#3
1.48μ
1.7μ
25°C
80°C
100°C
130°C
160°C
200°C
230°C
270°C
310°C
350°C
400°C
TRANSMISSION (RELATIVE SCALE)
1.0
1.5
2.0
2.5
WAVELENGTH (MICRONS)

Fig 8.