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Dielectric properties of Apollo 14 lunar samples

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Abstract—Laboratory characterization of dielectric properties of lunar samples 14301,41, 14310,75, 14318,30 and 14321,163 is made. Our measurements of dielectric constants and losses were made over a range of frequency from 100 Hz to 10 MHz and temperature from 77° to 473°K by two-terminal capacitance substitution methods. The dielectric behavior of these samples is generally similar to other lunar samples from Apollo 11 and Apollo 12 sites when these samples are free from absorbed moisture. As did sample 12002,58, sample 14310,75 showed a distinctive dispersion that may be associated with the presence of water, and different values of the activation energy for conduction as the temperature was varied. The activation energies range from about 0.03 to 0.5 eV. Values of the dielectric constant for lunar basalts like sample 14310 seem to run a few percent higher than those for terrestrial basalts with similar chemical composition. Our lunar samples contained metallic free iron and other high-conductivity materials in minute amounts; we believe our observed values of relatively high "apparent" dielectric constants for these lunar samples appear consistent with the presence of the high-conductivity materials in the samples.

INTRODUCTION

LABORATORY STUDIES on electrical properties of returned lunar samples from Apollo missions become of fundamental importance to the analysis of signals for interplanetary communications and also to the planning and subsequent interpretation of future in situ electrical experiments on the moon. For the last two years, since the receipt of Apollo 11 lunar samples and to date with Apollo 15, we have been concerned with laboratory characterization of electrical properties of returned lunar samples; much of our effort has been reported in various publications. As part of our continuing studies on the physical properties of lunar samples, we report in this paper laboratory measurements of dielectric constants and losses of lunar samples 14301, 14310, 14318, and 14321 returned from Apollo 14 mission.

Our measurements of dielectric constants and losses were made over a range of frequency from 100 Hz to 10 MHz and temperature from 77° to 473°K by two-terminal capacitance substitution methods. The methods have been described by von Hippel (1954) and are those adopted by the American Society for Testing Materials as the standard technique under ASTM Specification D150-68. Laboratory measuring equipment includes wide-range bridge devices, resonant cavities, and standing-wave equipment available at the MIT Laboratory for Insulation Research. A laboratory-built capacitance bridge covers a wide range of frequencies; the resolution is 0.002 pF or about 2% in dielectric constant and a few microradians in loss angle. Effects of some imperfect geometries of lunar samples and parallel capacitance of sample holders limit the accuracy of the dielectric constant values to about 5% and of the loss tangent measurements to about 10%.

LUNAR SAMPLES

The chemical and mineralogical description of samples 14301, 14310, 14318, and 14321 may be found in a report of the Lunar Sample Preliminary Examination Team (LSPET, 1971); readers are referred to this report for detailed information about these samples. A brief description of their character and density is made in Table 1.

Methods of transportation, handling, heating, and drying of lunar samples, along with other experimental details utilized in the present work were exactly the same as ones described by Chung *et al.* (1970, 1971), and this same description will not be repeated here. In the present work, our special attention was given to drying and baking of lunar samples at 570°K in vacuum oven so that our samples would be free from absorbed moisture.

DIELECTRIC PROPERTIES OF LUNAR SAMPLES

Values of the dielectric constant κ', loss tangent, and conductivity for sample 14321,163 are shown in Figs. 1 through 3. We note the very flat frequency-dependence of κ', indicating the successful minimization of water contamination as discussed earlier by Chung *et al.* (1971) and Katsube *et al.* (1971). In their measurements of κ' for some terrestrial basalts, Saint-Amant and Strangway (1970) made similar observations. The effects of absorbed water on κ' were discussed by Chung (1972). In characterizing the dielectric constant spectra, it is convenient to define the "Percent Frequency Effect (PFE)" as:

$$\text{PFE (in \%)} = 100(\kappa'_{10^2} - \kappa'_{10^6})/\kappa'_{10^2} \quad (1)$$

where κ'_{10^2} and κ'_{10^6} refer to the measured κ' at 10^2 and 10^6 Hz, respectively. Large values of PFE imply large dispersion at low frequencies; for lunar samples these

Table 1. Lunar samples and their material characteristics

Sample number	Density (gm/cm^3)	Classification	Characteristics
14301	2.30	Clastic	Feldspathic clasts with plagioclase as the major phase. Other phases seen are pyroxene, olivine, ilmenite, metallic iron and zircon crystals.
14310	2.85	Crystalline	Fine-grained crystalline rock with scattered small cognate inclusions. Plagioclase and anhedral pyroxenes are the major phases; about 70 volume percent plagioclase and a 30% clinopyroxene. Minor phases are ilmenite, troilite, metallic iron and some spinels with iron-rich compositions.
14318	2.30	Clastic	Feldspathic clasts, very similar to sample 14301.
14321	2.35	Fragmented	Complex texture showing a variety of rock and mineral clasts set in matrices. Anorthitic plagioclase and clinopyroxene are the major phases. Orthopyroxene, olivine, ilmenite, troilite, and metallic iron are the minor phases.

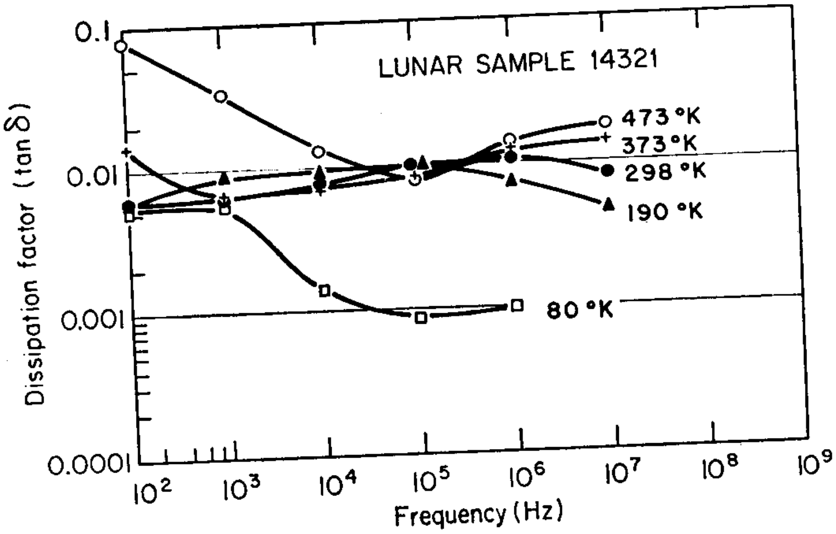

Fig. 1. Dielectric losses in sample 14321,163 as a function of frequency and temperature.

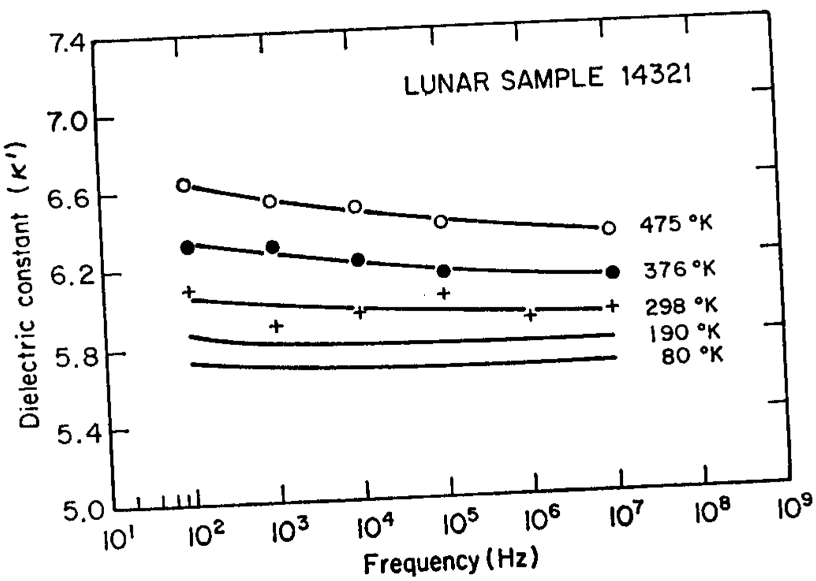

Fig. 2. Dielectric constant of sample 14321,163 as a function of frequency and temperature.

values are tabulated in Table 2. The flat frequency dependencies as observed earlier by Katsube and Collett (1971) on Apollo 11 and 12 lunar samples have PFE values in the range of 0 to about 17. Our earlier data on lunar samples containing small amounts of absorbed water show a PFE value in excess of 80%. With baking the lunar samples in a vacuum oven at 570°K for about 10 hours and then the measurement of κ' in the dry nitrogen atmosphere, the PFE values dropped down to 1 to 8%. As is seen in Table 3, sample 14321,163 has dielectric properties that are very similar to those of other lunar samples studied by Chung *et al.* (1972, 1971) and Katsube and Collett (1971).

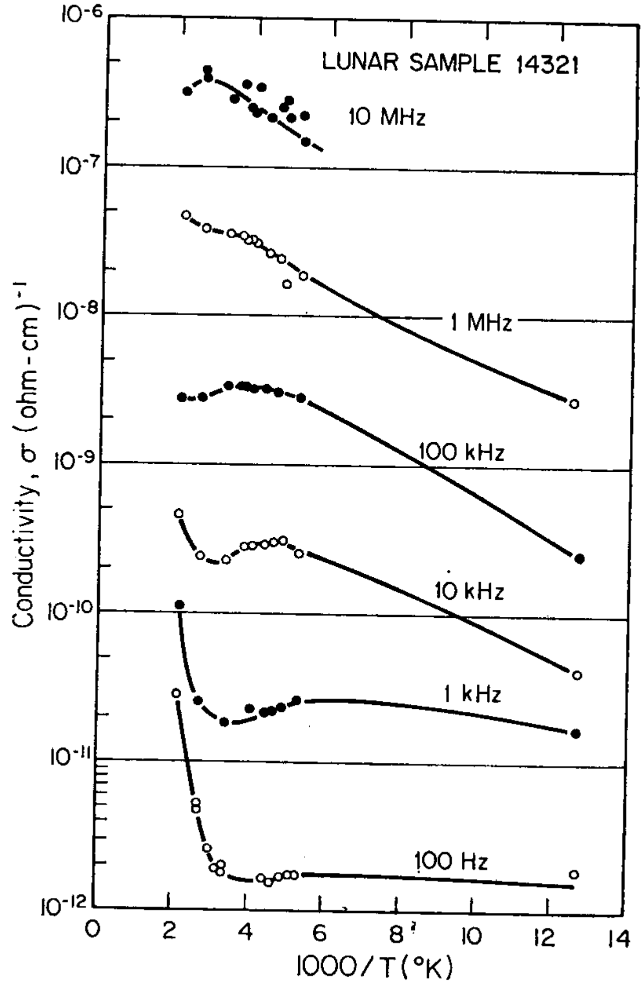

Fig. 3. Electrical conductivity of sample 14321,163 as a function of frequency and temperature.

Table 2. Extrema of loss tangents and percent frequency effect (PFE) of lunar samples.

Sample number	tan δ ($10^3 - 10^6$ Hz)	PFE* (%)	Sources and references
10020	0.1–0.4	82	Chung *et al.* (1970)
10046	0.06–0.2	79	Chung *et al.* (1970)
10017	0.02–0.09	0.3	Katsube and Collett (1971)
10065	0.02–0.1	17	Katsube and Collett (1971)
10084	0.01–0.11	0	Katsube and Collett (1971)
12070	0.005–0.13	3	Katsube and Collett (1971)
12002,84	0.02–0.05	6	Katsube and Collett (1971)
12002,58	0.03–0.05	30	Chung *et al.* (1971)
12022	0.2–0.3	81	Chung *et al.* (1971)
14301	0.03–0.06	8	This work
14310	0.007–0.02	7	This work
14318	0.025–0.06	8	This work
14321	0.008–0.01	1	This work

* High value of PFE may represent low-frequency dispersion associated with the presence of the absorbed water in the sample (see text).

Table 3. Dielectric constants and losses and conductivities of lunar samples and their comparison with some terrestrial materials.

Material and sample no.	Density (gm/cm^3)	T (°K)	Freq. (Hz)	κ'	tan δ	Conductivity T (°K)	Conductivity (mho/cm)	Reference*
CN Basalt-A	2.55	300	10^6	10.1	0.03	473–573	3×10^{-12}	1
CN Basalt-B	2.60	77–473	10^6	6	0.05	77–473	$\sim 10^{-10}$	2
Andesite Basalt-A	?	—	—	—	—	573–720	$\sim 10^{-6}$	3
Andesite Basalt-B	?	—	—	—	—	467–850	$\sim 10^{-6}$	4
Diabase	?	—	—	—	—	473–900	$\sim 10^{-6}$	4
LMT-J Basalt	3.01	77–473	10^6	6	0.002	77–473	10^{-10}	2
10020	3.18	77–473	10^6	9–16	0.002–0.3	278–473	10^{-10}	2
10046	2.21	77–250	10^6	9–13	0.09–0.2	77–250	10^{-10}	2
10017	3.10	300	10^6	8.8	0.075	300	4.2×10^{-10}	5
10065	2.45	300	10^6	7.3	0.053	300	6.7×10^{-10}	5
10084	?	300	10^6	3.8	0.0175	300	2.2×10^{-10}	5
12070	?	300	10^6	3.0	0.025	300	2.2×10^{-10}	5
12002,84	3.10	300	10^6	8.3	0.051	300	2.4×10^{-10}	5
12002,85	3.04	300	10^6	7.8	0.056	300	1.9×10^{-10}	5
12002,58	3.30	78–473	10^6	8–10	0.02–0.09	300–473	10^{-10}	6
12022,60	3.32	78–473	10^6	7–14	0.002–0.2	77–300	10^{-10}	6
14301,41	2.30	77–340	10^6	5	0.001–0.6	77–340	—	7
14310,37	2.17	300	10^6	4.8	0.05	300	1.5×10^{-11}	7
14321,163	2.40	77–376	10^6	6	0.001–0.15	77–200	—	7
14321,228	2.35	300	10^6	5.9	0.01	300	2×10^{-12}	7
14310,75	3.30	200–374	10^6	6	0.006–0.015	77–200	—	7
14310,75	3.30	200–374	10^6	7	0.006–0.015	200–300	—	7
14310,72	2.86	300	10^6	6	0.02	300	2×10^{-12}	7
14318,30	2.30	300	10^6	5.1	0.05	300	1.5×10^{-12}	7

* References: (1) St. Amant and Strangway (1970). (2) Chung, Westphal, and Simmons (1970). (3) Noritomi (1961). (4) Parkhomenko (1967). (5) Katsube and Collett (1971). (6) Chung, Westphal, and Simmons (1971). (7) This work.

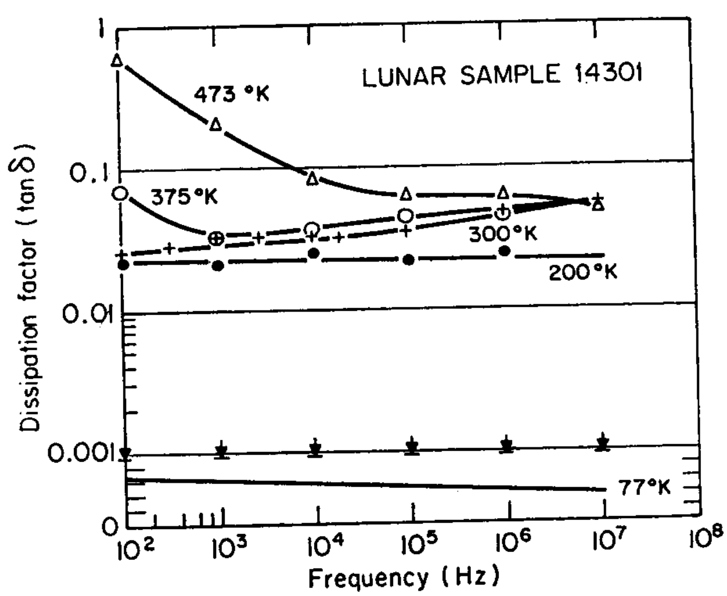

Fig. 4. Dielectric losses in sample 14301,41 as a function of frequency and temperature.

The dielectric spectra measured on sample 14301,41 are shown in Figs. 4 through 6. We observed a slight dispersion at low frequencies as is shown in Fig. 5. We also observed a Maxwell-Wagner type relaxation at high frequencies and high temperatures. Limited work on sample 14318 shows almost the same dielectric spectra as those observed for sample 14301. The dielectric properties measured on sample 14310,75 are shown in Figs. 6 through 10. The sample shows a strong relaxation effect of the Maxwell-Wagner type; an analysis of this relaxation is made in Fig. 11.

From the dielectric theory of solids, we have (see, for example, Poole and Farach, 1971)

$$\kappa = \frac{\varepsilon}{\varepsilon_0} = \kappa' - i\kappa'' = \kappa_\infty + \frac{\kappa_0 - \kappa_\infty}{1 + (i\omega\tau_0)^\lambda}; \qquad \lambda = (1 - \alpha) \tag{2}$$

where all the terms are exactly as described by Poole and Farach (1971); we have neglected contributions from the dc conductivity effects. For a single relaxation of the Debye type, the coefficient α is zero. An analysis of our data on sample 14310 in accordance with equation (2) is shown in Fig. 11, and we obtained a value of $\alpha = 0.345$ to give the best fit to our data. The single point at 100 Hz does not fit the Cole-Cole plot of Fig. 11; we believe that either a second distribution which may be appearing at lower frequencies, as noted by Khalafalla (1971), or an effect arising from the electrode contributions to the dispersion at this frequency range is associated with this datum point.

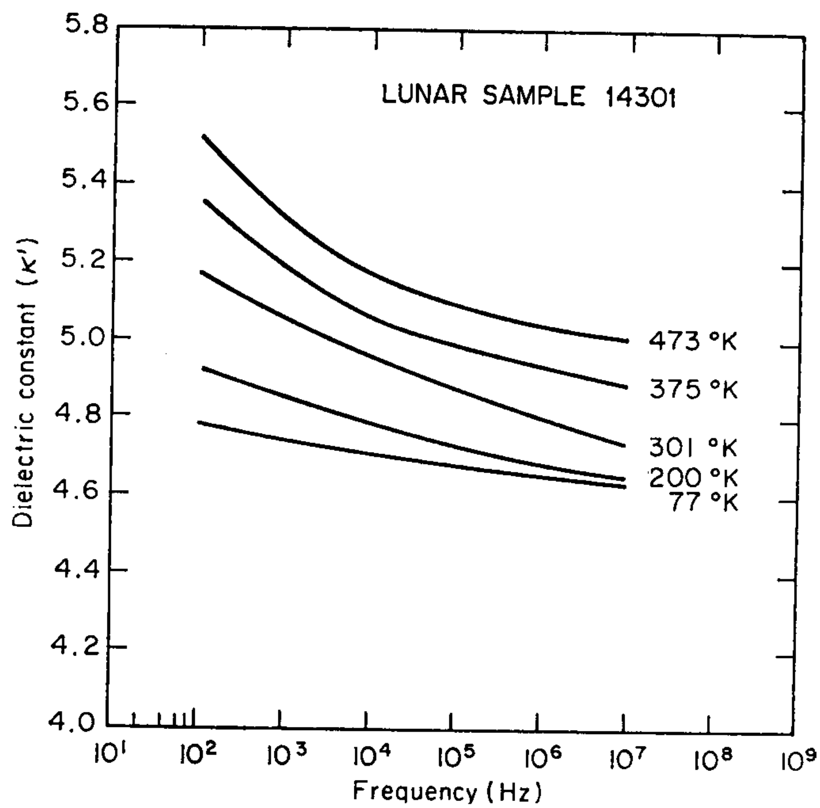

Fig. 5. Dielectric constant of sample 14301,41 as a function of frequency and temperature.

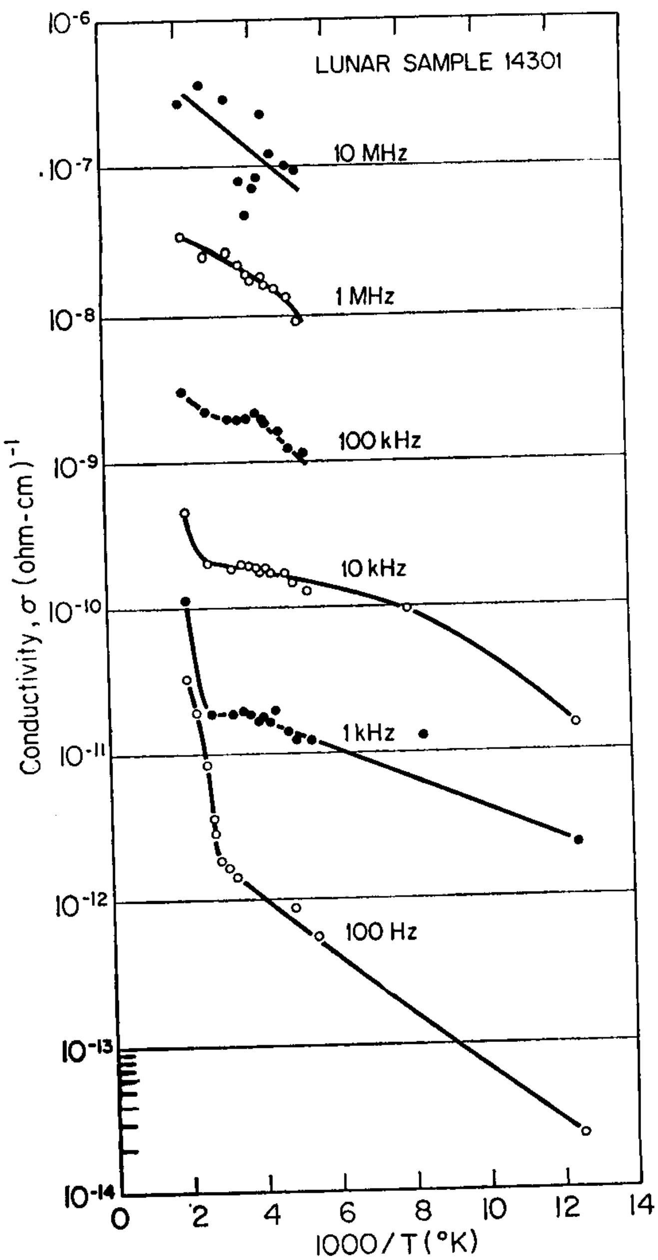

Fig. 6. Electrical conductivity of sample 14301,41 as a function of frequency and temperature.

An expression for loss tangent corresponding to equation (2) is

$$\tan \delta = \frac{\kappa''}{\kappa'} = \frac{(\kappa_0 - \kappa_\infty)(\omega\tau_0)^{1-\alpha} \sin (1 - \alpha)(\pi/2)}{\kappa_0 + (\kappa_0 + \kappa_\infty)(\omega\tau_0)^{1-\alpha} \cos (1 - \alpha)(\pi/2) + \kappa_\infty(\omega\tau_0)^{2(1-\alpha)}} \tag{3}$$

where ω is the frequency and τ the relaxation time. When $\alpha = 0$, equation (2) reduces to the well-known equation of the Debye case, and when $\alpha = 1$, equation (3) results in an expression describing a frequency-independent, constant loss tangent. Thus, for sample 14310,75, the relaxation frequency is spread over a range of τ (about the τ_0 point) as implied by $\alpha = 0.345$.

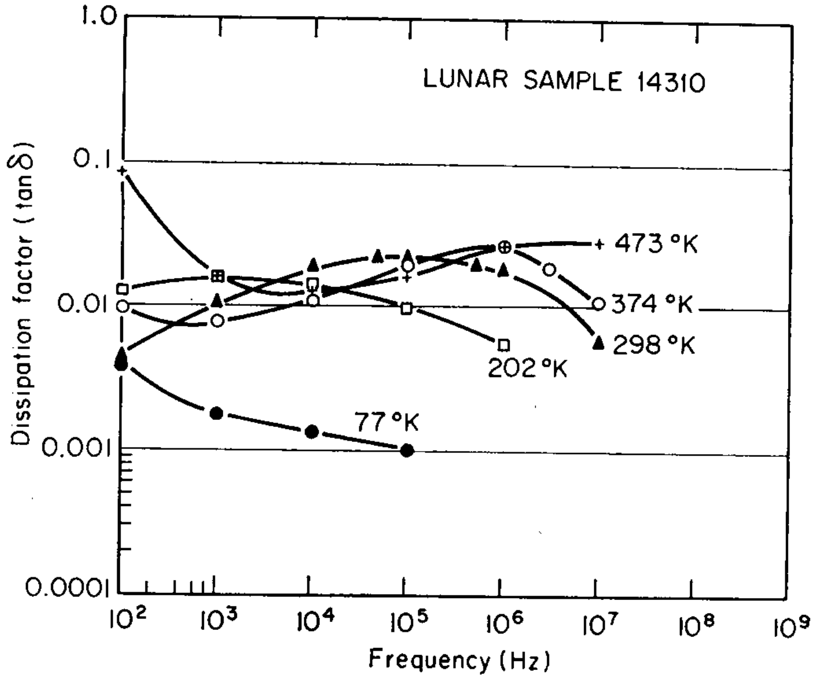

Fig. 7. Dielectric losses in sample 14310,75 as a function of frequency and temperature.

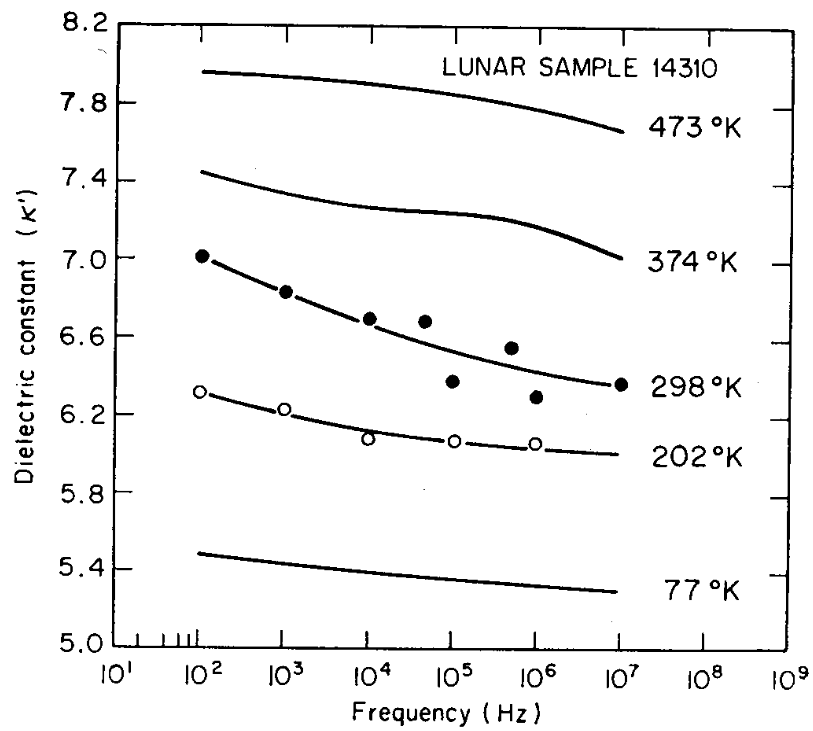

Fig. 8. Dielectric constant of sample 14310,75 as a function of frequency and temperature.

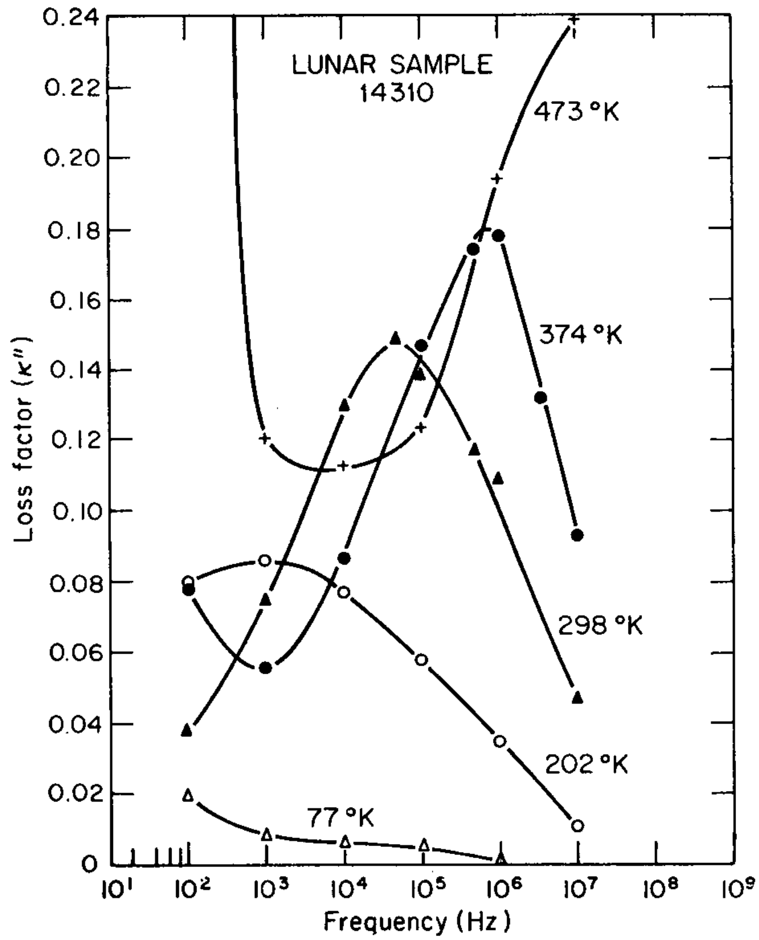

Fig. 9. Loss factor κ'' of sample 14310,75 as a function of frequency and temperature; illustration of the Maxwell-Wagner type relaxation due possibly to H_2O residue.

The loss tangent shown in Fig. 9 is typical of the dielectric losses seen in most igneous lunar basalts we studied. The activation energies associated with these relaxations can be estimated either from the temperature dependence of the frequency of peak loss, or from the peak value of κ'' varying with temperature in the usual way (see, for example, Saint-Amant and Strangway, 1970). For sample 14310,75 we estimate that the activation energy E_0 is in the neighborhood of 0.03 eV as obtained from the temperature dependence of κ'' data. The dielectric E_0 calculated from the temperature dependence of the relaxation frequency as inferred by the peak loss is in the range of about 0.2 to 0.5 eV.

The dielectric constants and losses, conductivities, and the activation energies for the lunar samples studied thus far are summarized in Table 4, and a comparison with the properties of similar materials of terrestrial origins is made in the table. An attempt to correlate the measured properties with the chemical composition of lunar samples is made also in Table 4; as is seen from this table, however, no generalization can be made at this time.

In summary, then, the following observations are listed:

(1) The dielectric behavior of samples 14301, 14310, 14318, and 14321 is generally similar to other lunar samples from Apollo 11 and Apollo 12 sites when these samples

Table 4. Electrical parameters of lunar samples and their possible relation to concentration of high-conductivity phases (all the parameters are evaluated at 300°K).

Sample no.	Sample classification	FeO (mole %)	TiO_2 (mole %)	Ilmenite (mole %)	κ'	tan δ	E_0(diel) (eV)	E_0(cond) (eV)
10020	Crystalline	19.35	10.72	15	11	0.15	—	0.22
10046	Breccia	19.22	10.35	9	13	0.07	—	—
10017	Crystalline	19.79	11.82	15–20	8.8	0.075	—	—
10065	Breccia	—	—	—	7.3	0.053	—	—
10084	Fines	15.62	7.5	12–14	3.8	0.0175	—	—
12070	Fines	17.0	3.1	3–6	3.0	0.025	—	—
12002,84	Igneous	13.0	4.3	3–4	8	0.05	—	—
12002,58	Igneous	13.1	3.9	4	8	0.05	0.04	0.23
12022	Crystalline	22.0	5.1	9–11	7–14	0.2	—	0.16
14301	Clastic	9.8	1.7	2	4.8	0.05	—	—
14321	Breccia	13.0	2.4	4	5.9	0.01	0.07	—
14310	Crystalline	7.7	1.3	2	6.4	0.02	0.3	0.24

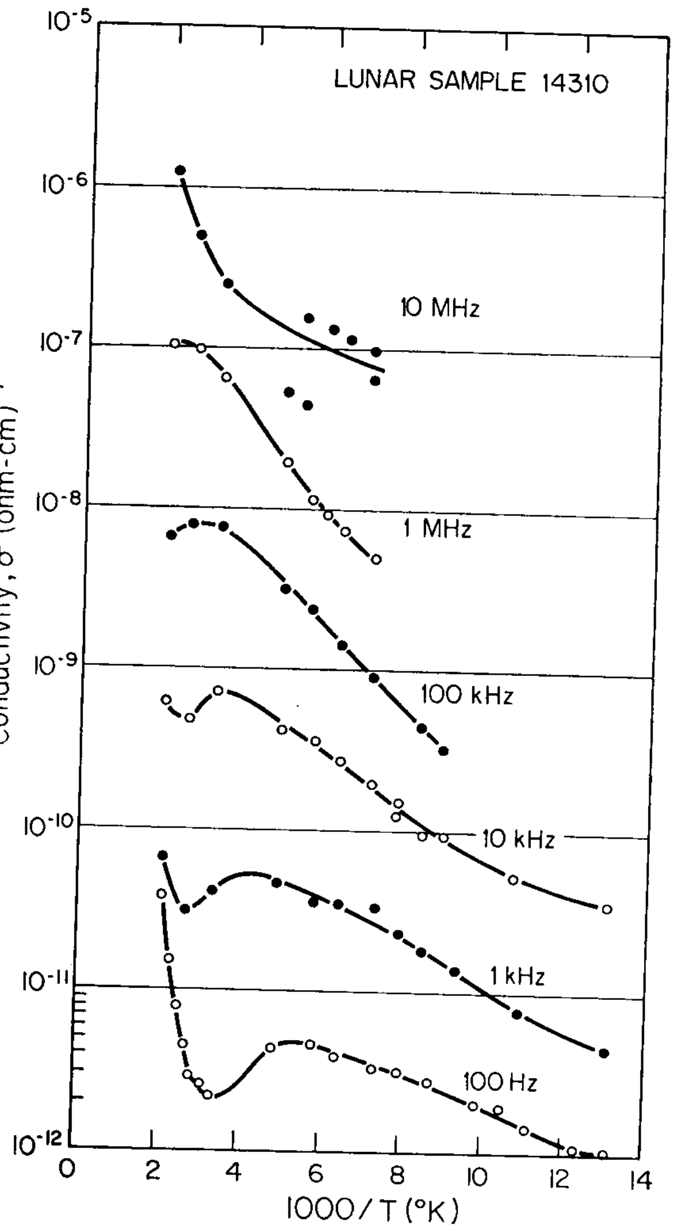

Fig. 10. Electrical conductivity of sample 14310,75 as a function of frequency and temperature.

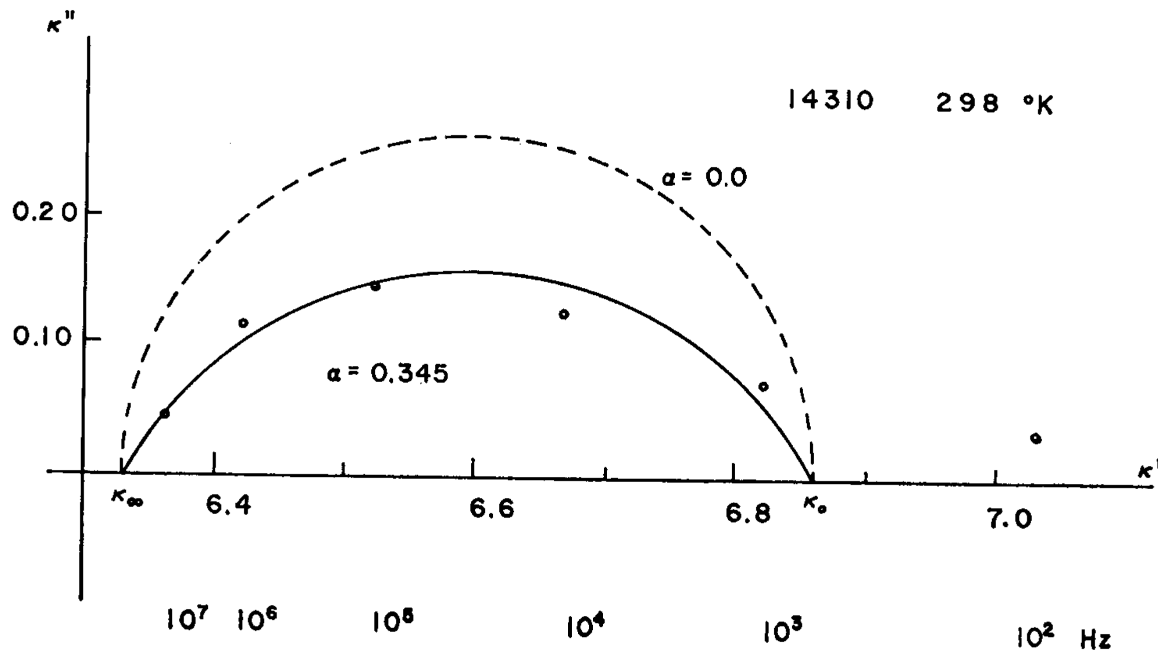

Fig. 11. Cole-Cole plot of sample 14310,75.

are free from absorbed moisture. As did sample 12002, sample 14310 showed a distinctive dispersion which may be associated with the presence of water, and different values of the activation energy as the temperature was varied. The activation energies range from about 0.03 to 0.5 eV and they seem to increase with increasing temperature.

(2) An abrupt change in the activation energy between 200 and 300°K from 0.02 to 0.3 eV, as seen in the conductivity versus temperature, is observed for all the lunar samples we studied. The mechanism of this change is not understood at the present time; it is suggestive of freezing effects of residual water vapor.

(3) The dielectric properties of the lunar samples fall within the range of terrestrial basalts; the lunar samples, however, exhibit slightly lower activation energies for conduction and slightly higher losses than the terrestrial basalt.

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