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# An Investigation of the Thermal Shock Resistance of Lunar Regolith and the Recovery of Hydrogen From Lunar Soil Heated Using Microwave Radiation

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## Abstract

The objective of this project is to develop a better understanding of the thermal shock properties of lunar regolith sintered using 2.45 GHz electromagnetic radiation and to do a preliminary study into the recovery of bound hydrogen in lunar soil heated using 2.45 GHz radiation. During the first phase of this work lunar simulant material was used to test whether or not microhardness data could be used to infer thermal shock resistance and later actual lunar regolith was used. Results are included on the lunar regolith since this is of primary concern and not the simulant results. They were similiar, however. The second phase of this work investigated the recovery of hydrogen from lunar regolith and results indicate that microwave heating of lunar regolith may be a good method for recovery of bound gases in the regolith.

## Introduction

In the first phase of work, we wished to show that a simple hardness test could be used to describe the thermal shock properties of lunar regolith thus avoiding costly time consuming mechanical property measurements. First through it was necessary to validate the premise that material hardness is related to the fracture toughness of the material. Palmquist[1] originally put forth the idea that there is a relationship between the extent of cracking that emanates from a microhardness indentation in a brittle material and that materials fracture toughness. It is known that the fracture toughness of a brittle material will remain the same or slightly increase up to the thermal shock temperature and then abruptly fall off. Further work showed that material hardness is related to the fracture toughness of that material and the relationship is shown in Equation (1).

$$H = \frac{\beta^2 EP}{K_c^2 \, C_o^3} \tag{1}$$

where

- $\beta$ = a material independent constant for Vickers induced radial cracks
- $\beta$ = 0.16 ± .04 for brittle materials
- E = Young's modulus
- H = material hardness

P = indentation load

$C_o$ = radial crack length

From Eq. (1) it is expected that material hardness will increase or stay constant up to the thermal shock temperature at which time it will abruptly decrease due to the decrease in Young's modulus. As the number of microcracks increase E is expected to decrease thus causing a decrease in hardness. This approach has been used to examine the fracture toughness of SiC whisker reinforced $Al_2O_3$ and we have used it to determine the thermal shock properties of Yttria-2 w% Zirconia composite material.

Microhardness data has been obtained on lunar regolith material which was first sintered at 2.45 GHz. Hardness measurements were performed using a LECO M-400 digital microhardness tester. Figures 1-3 show how material hardness behaved as a function of thermal shock temperature. Apollo 15 and 16 samples (which were crystalline) showed an increase in hardness up to the thermal shock temperature and then showed an abrupt fall off while the glassy Apollo 11 sample showed a steady decrease in hardness with increasing thermal shock temperature. Figure 4 shows typical microhardness indentations on an Apollo 15 sample. The curves of Figures 1-3 were obtained by averaging many data points for each thermal shock temperature.

More work needs to be done to relate observed thermal shock to the microwave sintered microstructure. There does appear to be an increase in thermal shock resistance due to the sintering in a 2.45 GHz electromagnetic field. At present individual grains of simulant Apollo 11 material are being examined using TEM to determine the structure of the grain surface. Initial results indicate that the outer 500 Å of a typical 1 micron size grain is amorphous while for the same material conventionally sintered the entire grain is crystalline. If this holds true for the surface of closed pores then the enhanced thermal shock resistance may be due to the hindrance of each pore to the formation of microcracks during the thermal shock test. Usually microcracks originate at pore surfaces during thermal shock. Also mechanical properties should be different if grain boundaries and a large region between grains are amorphous because viscous flow will dominate in the region between the grains while conventional brittle fracture mechanics will dominate within each crystalline grain.

One final topic should be addressed before proceeding on to a discussion of Task 2. This is a relative comparison of energy requirements to process lunar soil when it is heated using 2.45 GHz radiation versus conventional $10^{13}$ Hz radiation (IR heating). An apprxomation of the energy denstiy required to heat lunar soil may be made using data previously obtained.[2] An error in reference 2 is the ordinate of the power density curves should be cal/sec-cc not as shown cal/cc. Using the information in Figure 3 of reference 2 showing power density in a lunar soil for an electric field intensity of 300 V/cm in the soil one arrives at a power density of 400 cal/sec-cc to maintain a sample temperature of 1200°C. Heating the sample for thirty minutes (1800 seconds) to fully densify the lunar soil then requires (400 cal/sec-cc) (1800 sec) or $7.2 \times 10^2$ k cal/cc. Converting this to kw hr we obtain a value of 0.837 kw hr/cc of lunar soil. When compared to conventional heating significant energy savings will be achieved since heating rates of many thousands of degrees per hour can be achieved when microwave heating; however, depending on the thermal processing step conventional heating rates for oxide materials are limited to only 100-200°C/hr. Also microwave energy is deposited directly to the object being heating while in conventional heating, much of the energy is lost in heating the surrounding material. If we assume just the heating of lunar soil, then perhaps a solar furance may be more efficient than microwave heating; however, the material will require a much longer time to achieve a uniform temperature throughout the material.

In Task 2 a preliminary investigation into the recovery of bound hydrogen in lunar regolith was carried out using 2.45 GHz radiation to heat the regolith. In this work a 1.5 gram sample of Apollo 15 and Apollo 16 regolith material was heated in an enclosed pyrex sample holder through which first a helium carried gas was passed and later in the Apollo 16 work an Argon carrier gas was used. According to the work of Epstein and Taylor[3] hydrogen starts to be released from lunar regolith at around 500°C and continues until around 700°C. The amount of hydrogen released is of the order of 1 µg/gram of sample. In this work very sophisticated equipment is usually used such that extremely small quantities (of the order of 1 µg) of sample may be used. For our work the gas chromatograph used required a much larger sample (of the order of 1 gram or more). The model used in this work is a Varian Gas Chromatograph Model 90P-3 manufactured in 1966.

Since the lunar samples used for this work had been used for a prior experiment in which water and carbon material was used, each sample was analyzed at Los Alamos National Laboratory as to the amount of carbon present. Sample number 6999.75A (Apollo 16) had 6.8 ppm carbon; sample number 15999.126A (Apollo 15) had 69.5 ppm carbon; and sample number 10089.4A (Apollo 11) had 186.4 ppm carbon. For this work we used only a sample of Apollo 15 and one of Apollo 16 since they had much less carbon than did Apollo 11.

In gas chromatography a carrier gas is passed over one side of a wheatstone bridge while the carrier plus sample is passed over the other side of the bridge. Since the wheatstone bridge is heated to approximately 250°C any change in gas thermal conductivity will cause a change in bridge temperature. This change will be reflected in a change in electrical resistance. It is then possible to determine the amount and composition of an unknown gas in a sample by this technique. It was decided to first use as a carrier gas helium since the only known gas with a higher thermal conductivity is hydrogen. Everything else that might be released will then yield a positive peak while only hydrogen will yield a negative peak. This experiment was performed using a 1.5 gram sample of Apollo 15 material and the results are shown in Figure 5. Hydrogen release appears to occur at a lower temperature; however, in all probability the actual grain or intergranular temperature is much higher than the observed temperature. This is explained in a previous paper by Meek.[4]

In the next experiment Argon was used as a carrier gas. The resultant curve (Figure 6) yielded what appears to be a hydrogen peak in about the same temperature range as the previous experiment. Also as is shown in Table 1 only methane and ethane would show a negative peak and those peaks would be very small. The presence of water would not be detected using Argon while with He it would yield a positive peak. The other most likely gas to be released in these samples would be $CO_2$ and in Argon it again would be almost nondetectable. It thus appears that probably the strongly negative peaks observed in both of the experiments appear to be hydrogen. The Argon data were analyzed and indicate that much more hydrogen is released than was reported by other researchers, on the order of 70 µg of hydrogen per gram of Apollo 16 regolith. This may be due to the manner in which the 2.45 GHz radiation couples to the regolith or it may be due to error in our interpretation of the data. Clearly more work needs to be done to substantiate the preliminary data obtained above. If further work in this area is done, it may be worth while using a different carrier gas namely neon. The cost of neon may be prohibitive; however, as seen in Table 1 it is intermediate between He which gives a small hydrogen peak and Argon which yield a very large negative peak.

Table 1. Response Factor for Some Carrier Gases and Some Sample Gases.

| | Thermal Conductivity | Response Factor $1-\frac{\lambda_s}{\lambda_c}$ | | |
|---|---|---|---|---|
| Gas | (Cal/sec cm$^2$ °C/cm) x $10^{-6}$ | He | Ar | Ne |
| He | 360.36 | | | |
| Ar | 42.6 | | | |
| Ne | 115.71 | | | |
| $H_2$ | 446.32 | -0.24 | -9.5 | -2.85 |
| $CO_2$ | 39.67 | +0.89 | +0.07 | +0.66 |
| $H_2O$ | 42.57 | +0.88 | ~0.0 | +0.62 |
| $CH_4$ | 81.83 | +0.77 | -00.91 | +0.30 |
| $C_2H_6$ | 51 | +0.84 | -0.19 | +0.56 |

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3. S. Epstein and H. P. Taylor, Proceedings of Apollo 11 Lunar Science Conference, 1970, 1085.

4. T. T. Meek, "Proposed Model for the Sintering of a Dielectric in a Microwave Field," Journal of Materials Science Letters, 6, 1987, 638.

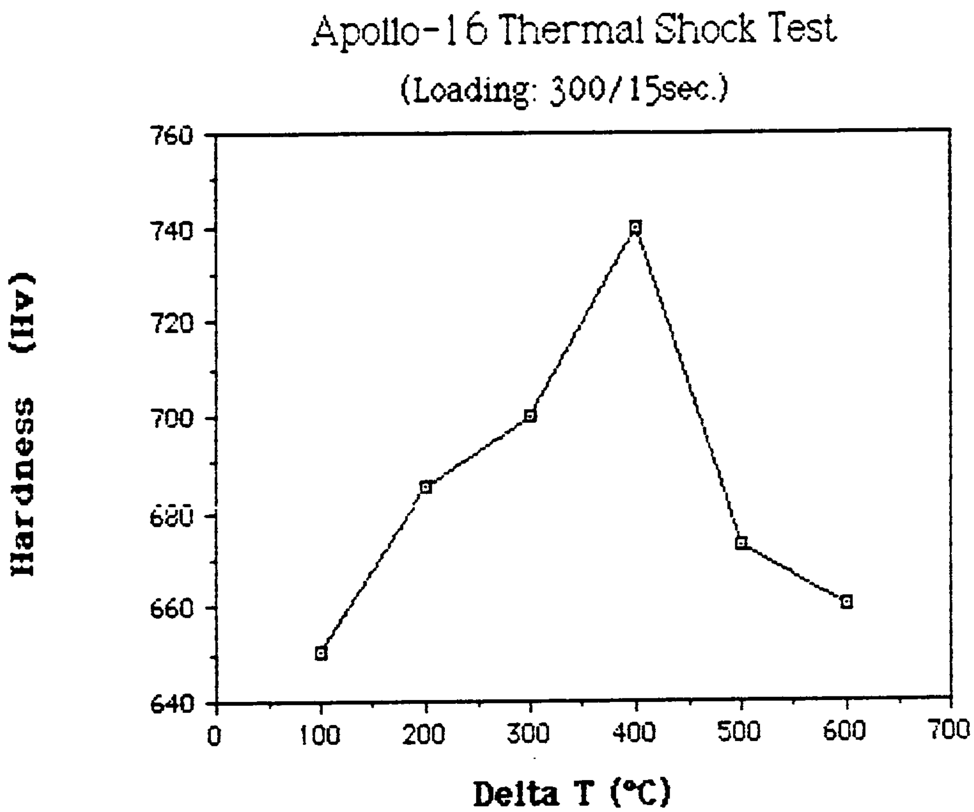


Figure 1. Microhardness vs. Delta T (°C) data for Apollo 16 soil. $\Delta T_C = 400°$. Hardness is in $kg/nm^2$.

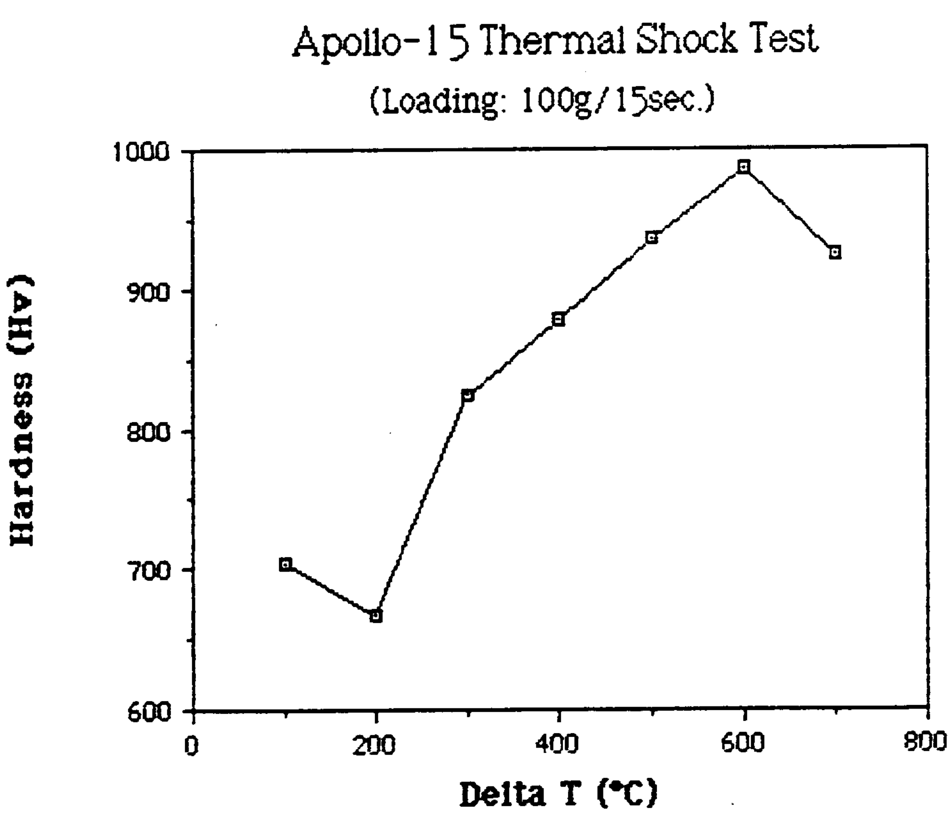


Figure 2. Microhardness vs. Delta T(°C) data for Apollo 15 soil. $\Delta T_C = 600°C$. Hardness is in $kg/nm^2$.

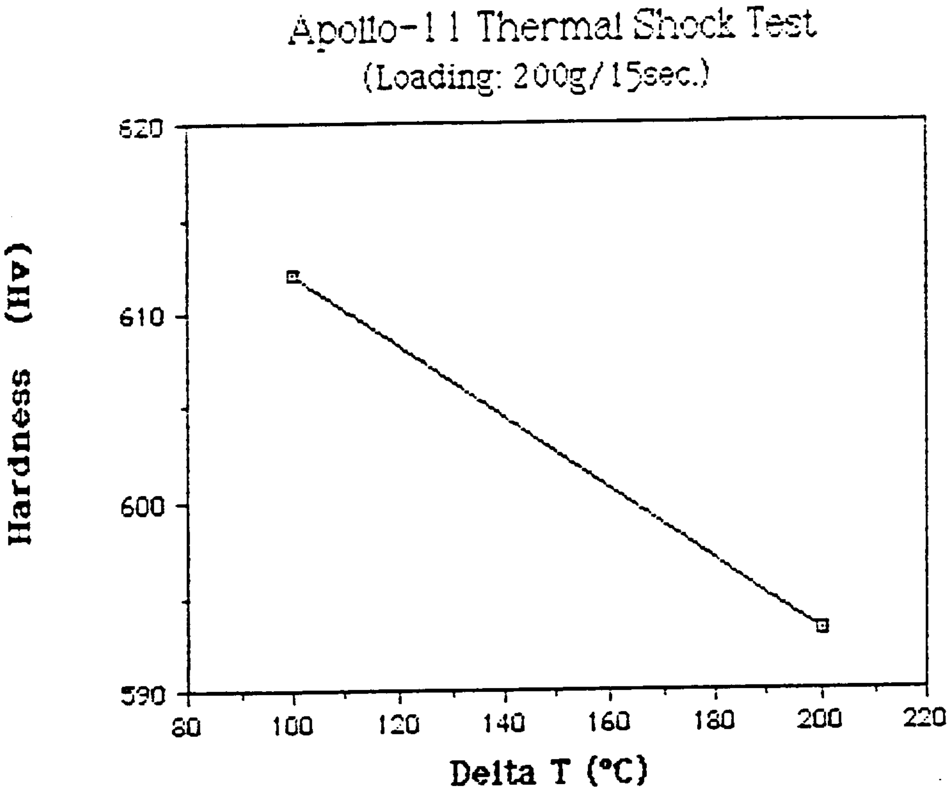


Figure 3. Microhardness vs. Delta T (°C) data for Apollo 11 soil. $\Delta T_C$ = 100°. Hardness is in kg/nm$^2$.

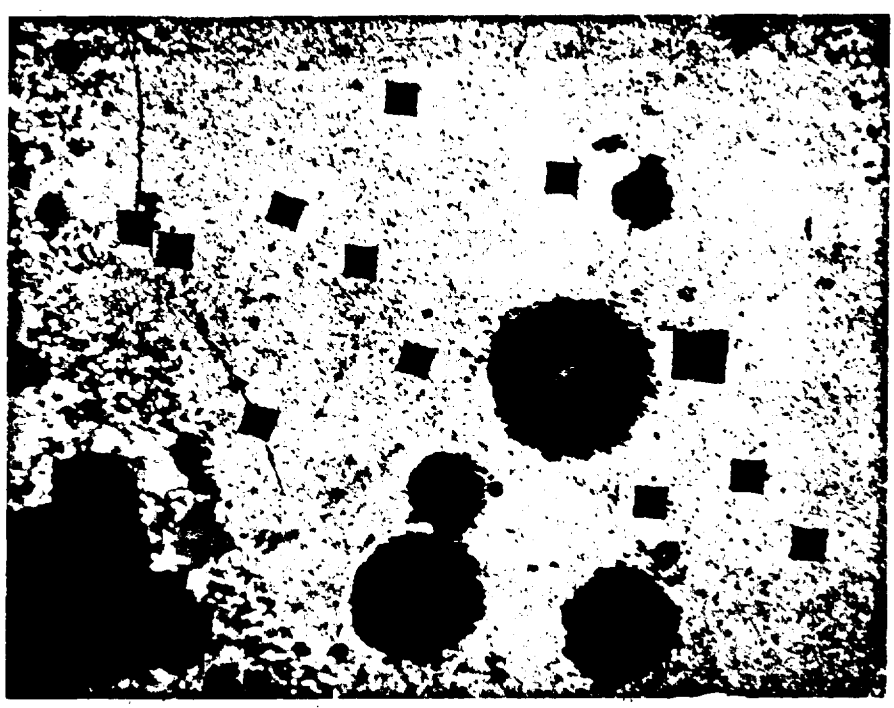

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Figure 4. Typical microhardness indentaions on an Apollo 15 sample sintered using 2.45 GHz radiation..

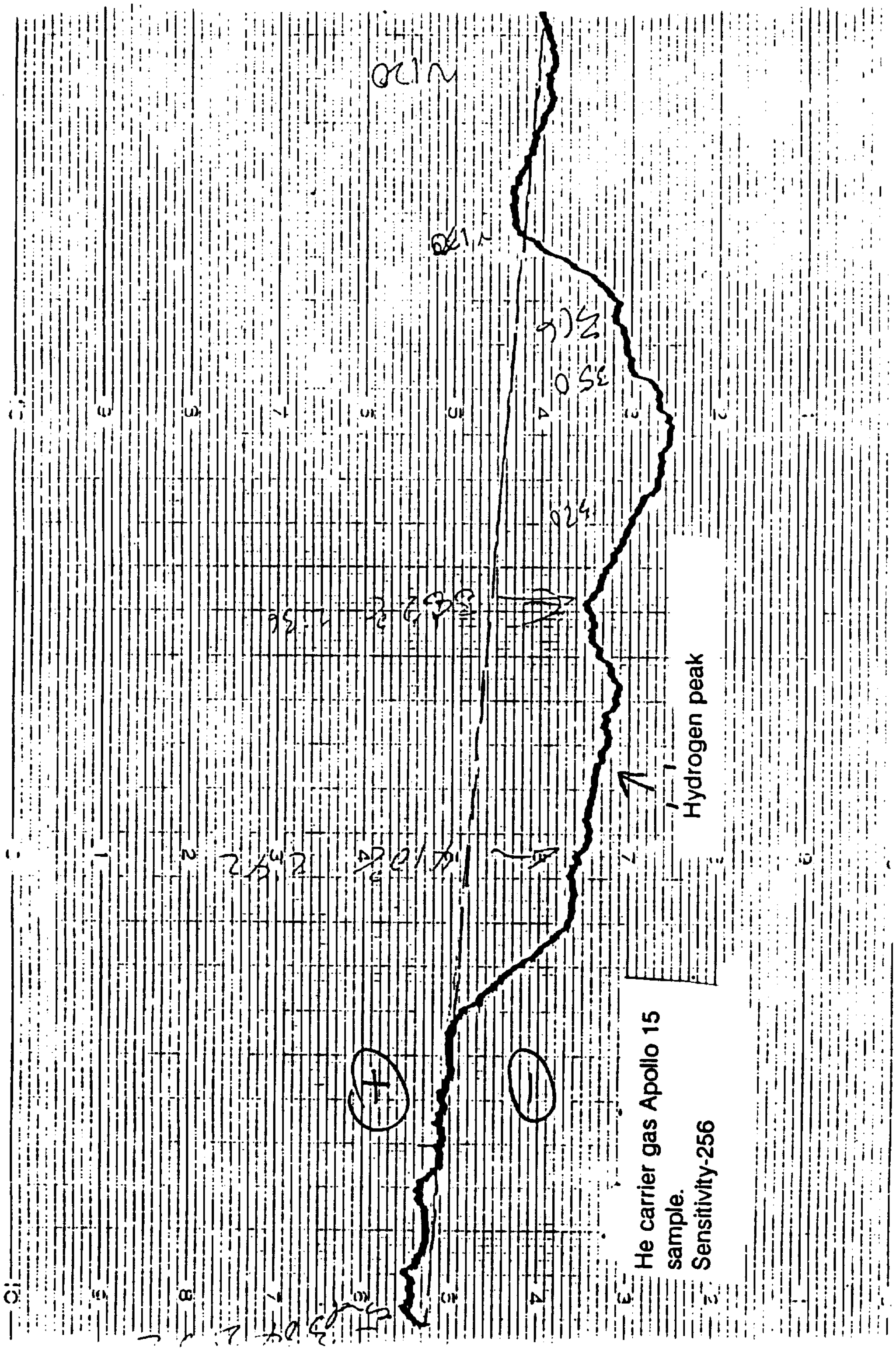


Figure 5. Gas chromatograph of hydrogen release in Apollo 15 soil heated using 2.45 GHz radiation. Carrier gas is He.

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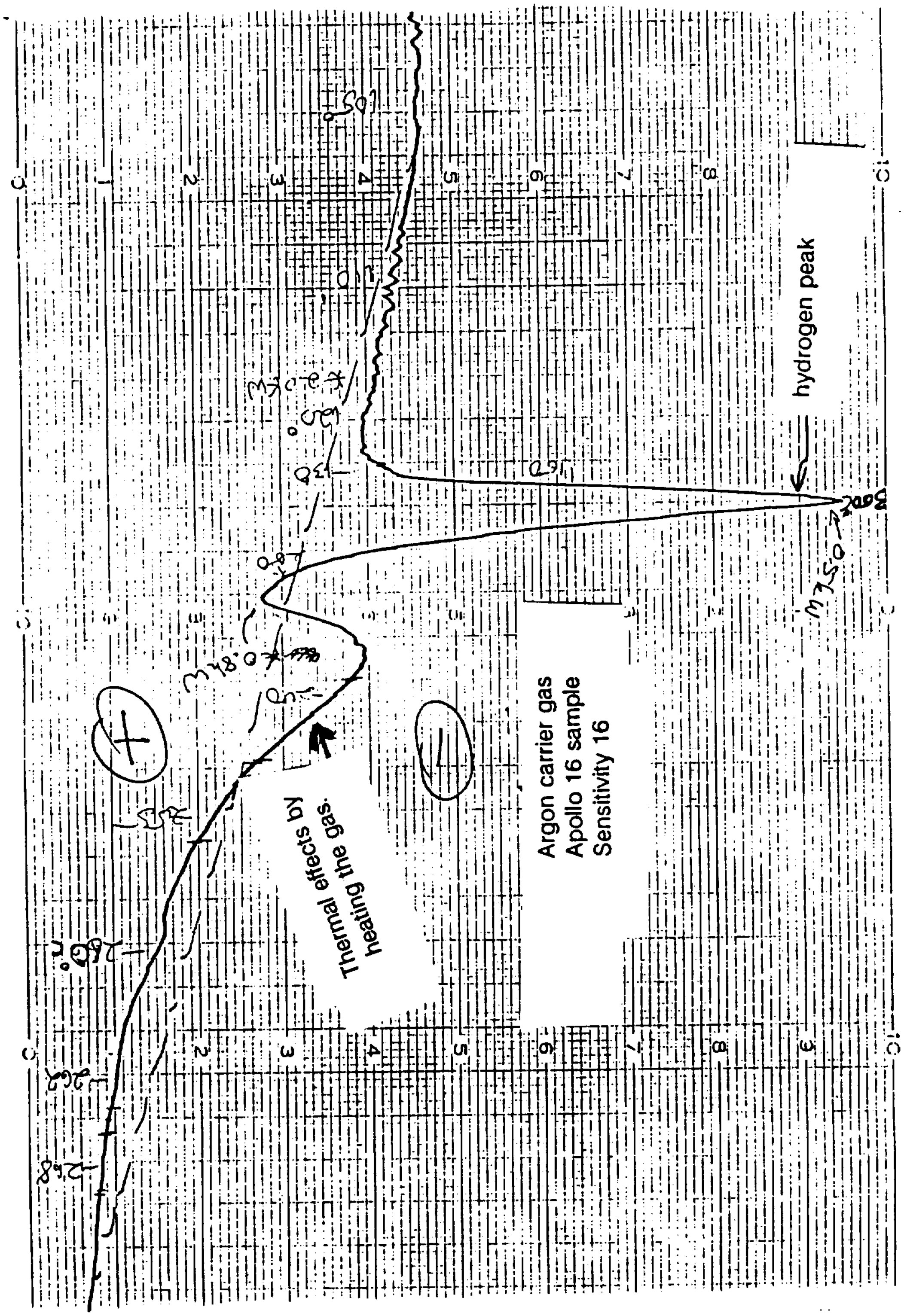


**Figure 6.** Gas chromatograph of hydrogen release in Apollo 16 soil heated using 2.45 GHz radiation. Carrier gas is Argon.

# B. REDUCTION OF CARBON DIOXIDE