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FINAL REPORT

MATERIALS COMPATIBILITY WITH THE VOLCANIC ENVIRONMENT

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## Materials Compatibility with the Volcanic Environment

The tasks outlined in the grant were the following:

- a) Expose metal samples in lava when available and determine the nature of the chemical attack.
- b) Determine the temperature and other properties of the lava.
- c) Expose samples supplied by Sandia to volcanic gases in fumaroles and drill holes and determine the nature and extent of the chemical attack.
- d) Measure temperatures and gas compositions in selected fumaroles and gases being evolved from lava.
- e) Cooperate with Sandia personnel in making geochemical and geophysical field measurements and tests on lava flows, lava lakes, and fumaroles when conditions permit such tests.
- f) Carry out laboratory tests to supplement field experiments.

Various progress reports have been submitted and this report will include only the recent results and final thoughts on the project.

### Corrosion of Metals and Alloys in Lava.

A major effort was made to cooperate with a research team from Sandia Laboratories to run materials compatibility, volcanic gas collection, and heat transfer experiments during the September 13-October 1, 1977 eruption of the Kilauea Volcano along its east rift zone.

Nine alloy sample strings were exposed but only four were recovered and those were taken back to Sandia by Dr. P. Modreski. Some preliminary results obtained from those samples are included in Appendix A. The four sets of recovered samples were the following:

String No. 1. Sixteen samples were strung on a 304 stainless steel cable reinforced with nichrome wire. Glass tubings were used as spacers. These samples were exposed for 18 hours (9/23/77-9/24/77) to hot gases at approximately 900°C emitting from a spatter cone. Eruption had occurred from the cone 3 days earlier, but the interior of the cone was still glowing hot, and at night the hot gases emitting from the cone glowed like flames from an open furnace indicating that the cone probably formed an outlet for the degassing magma.

String No. 2. Six metal samples were strung on a 304 stainless steel cable with ceramic spacers and the string was hung for 24 hours in a hot crack in solidified lava. The lava was from a 2-day old flow and it now formed a bank for the lava river as the direction of the flow had changed. The surface of the solidified lava was black but it was still hot enough to burn the soles and heels of boots whereas the interior of cracks, about 5-10 inches wide and 2-5 feet deep glowed red hot. The temperature within the above crack was 850°C.

String No. 3. Seven samples were strung on a 304 stainless steel cable with short lengths of Nichrome wire twisted around the cable to act as spacers. This string was exposed in the lava river on 9/30/77 for about 10 minutes. The temperature of the lava river was 1080°C. The surface of the lava river was a mixture of fluid, flowing molten rocks and black, broken slabs of recently solidified lava flowing quite rapidly. At this fourth eruption stage, HVO estimated that the fountains were up to 100 m high and the discharge rate was about  $5-7 \times 10^3 \text{ m}^3/\text{hour}$  and the lava advanced towards SE at up to 300 m/hour. It seemed that the velocity of the lava river was faster than the above estimate, possibly because the experiments were conducted closer to the eruption site. Unfortunately, the group was preoccupied with other matters and forgot to measure the lava flow rate. The string was thrown onto the surface of the river and the samples were sucked down below the surface. It was very difficult to pull the samples out even after the short exposure time, and if they had been exposed for longer periods, the samples would have been lost because the string would have been sucked deeper into the flow.

String No. 4. Sixteen different alloys were strung on a 304 stainless steel cable using ceramic spacers. The string was exposed of 6 hours on 10/2/77 in a hot crack in a cooling lava riverbed after the eruption had ceased. The temperature decreased from 840°C to about 600°C at that location during that period.

Preliminary results obtained from the recovered samples showed that Fe, Ni, and Fe-Ni alloys were the most heavily oxidised. The Mo and W alloys showed some attack and only negligible reaction was seen on 310 stainless, Hastelloy-C, Inconel 600, Inconel 718, Rene-41, and on Nichrome. See also Appendix A. More detailed analyses of those samples have presumably been done by Sandia personnel but the U.H. group does not know the results.

The following strings were not recovered:

Strings No. 5 and 6. These samples were mounted on rods, about 12 mm dia and 1.2 m long. The strings were placed in a lava flow on 9/26/77 and could not be located later because subsequent lava flows had covered them completely

String No. 7. This string was prepared in the field and was placed in a lava flow spilling over from the main lava river. This string also was not found later.

String No. 8. In this case an attempt was made to fix the location where the samples were placed by connecting the samples to a long length of a 304 stainless cable which was then tied to two ohia trees about 1.8 m above the ground. This string also was lost because the flow at that site caused the samples to be buried under solidified lava over 2 m thick. Also, the ends of the cable which was fixed to the trees were not found.

String No. 9. Nine samples strung on a 304 stainless cable were immersed in the lava river close to its banks on 7/30/77. The ends of the cables were secured to a couple of heavy rocks behind the levee of the lava river. Unfortunately, no trace of the samples nor the cable itself could be found.

It can be concluded that the attempts to expose the samples to lava were not successful. The main reason was that none of the group had ever been at an eruption site before. In retrospect, it was unfortunate that the sample strings most suitable for exposure in the lava river, i.e., those mounted on rods, were wasted on side flows. At that time, there was considerable uncertainty as the eruption sites had changed several times and no one knew how long the current phase would last. Also, the group had been waiting for the opportunity to be at the eruption site for several years, thus there was urgency to begin the experiments immediately. After the lava achieved steady flow SE in its channel to offer an excellent opportunity for materials compatibility experiments, the available strings were those suitable for exposure in fumaroles. Clearly, the small, approximately 25 m.m. dia samples strung on stainless steel cables, about 6.5 m.m. dia are unsuitable for exposure in the lava river. The fact that the lava river literally swallowed the small samples thrown onto its surface (and also the approximately 50 mm dia. stainless steel pole used in the heat-transfer experiment) clearly indicated that complex currents existed within the lava river. At this time, there does not seem to be any work done on the nature of those currents. Several new techniques may be tried if the experiments can be repeated. Larger samples, about 30-50 mm disks may be mounted on about 10 m.m. rods and the rods can be tied to about 6 m.m. dia. stainless cables. The ends of the cables can be securely fastened to large boulders or trees at some distance from the lava flow. Some samples may also be immersed in stagnant pools of lava which are away from the main flow so that the samples will not be carried downstream nor be sucked deep into the pool. The pools should still be physically connected to the lava river so that it will remain molten, otherwise a thick crust of solidified lava on the surface may make it impossible to pull the samples out. A portable winch is a definite requirement to pull the samples out. It would be advisable to monitor the status of the samples such as by pulling on the cable intermittently to insure that the samples do not become stuck under ledges or other obstructions in the lava river or that the integrity of the stainless cables had not been corroded away. Further, at least a dozen strings should be prepared and exposed to insure that some will be recovered, and also to have samples exposed for different times.



## Volcanic Gases.

Materials used in geothermal projects will inevitably be exposed to corrosive gases incorporated in the magma. These gases have been studied since 1917 but the results are not completely satisfactory because of sample contamination by air, water vapor, etc. during collection. The gaseous composition is characteristically variable with location, and at a given location, with depth of collection and time since eruption. Also rainfall, burning vegetation, and airflow patterns increase complications. Thus it is necessary to determine both the gaseous compositions, and compositional variations at sites where corrosion samples are placed.

The early (1975) plans in this project were to collect the gas samples using the Naughton tubes and analyzing them at Hilo College (now the University of Hawaii, Hilo) using the gas chromatograph being built by Dr. Bruce Finlayson from equipment donated by Sandia Laboratories. Unfortunately, this arrangement could not be carried out as Dr. Finlayson emigrated to New Zealand. Plans were made for Dr. E. Herlicska, also from Hilo College to do the gas analysis. However, the necessary parts for the chromatograph (Beckman, GC-4, large oven, dual column) could not be found nor bought as Beckman service personnel said that the above unit was outdated and the two 300V batteries for the detector tube electronics were no longer available. Further, sufficient money was not available in the project to buy a new chromatograph. Thus, gas collection was done in response to requests from Sandia and the collected samples were sent to them for analysis. This arrangement was generally unsatisfactory as a number of tubes broke in transit. The collector was also was not promptly aware of the results and so improvements in both collection methods and laboratory experiments were tardy.

Various sampling tubes and techniques were tried in the course of this study.

1. Naughton tubes were used initially. These were 10 mm evacuated quartz tubes about 1 m long with a hook shaped at the sampling end. This hook was broken off after inserting the collection end inside a fumarole, spatter cone, or a suitable site at a lava flow. Activated silica gel in the tube prevented reaction between the gases, especially between sulfur dioxide and hydrogen sulfide. These tubes were expensive, cumbersome, and fragile and a number of them broke during handling and shipment.
2. Samples were also collected using a manual peristaltic pump, teflon or tygon tubing and small evacuated bottles. One end of the tube was lowered into the fumarole and the gases were sucked up using the pump. When condensation occurred in the tube indicating that the air originally within the tube had been exhausted, then a T-junction stopcock was opened to allow the gases to flow into the sampling bulb. After collection, these bulbs as well as the Naughton tubes were sealed using a portable oxy-propane torch. Gas contamination can occur at this time if a partial vacuum is created within the tubes or bulbs by the cooling gases. However, positive gas pressures within the tubes can be obtained by leaving the collection tubes in the vent for 10-15 minutes before retrieval. The results showed that air contamination during

sampling is more easily avoided by using the pump. Condensation of water in the tubing depletes the water content in the collected gases and will also change the concentrations of the other water soluble gases in the sample. This problem can be minimized by collecting the condensate and analyzing its contents.

The fumarolic gas compositions have been analyzed at Sandia by Modreski and Gerlach and the results obtained are shown in Appendix B.

3. At the 1977 eruption, the volcanic gases were collected using a 250 ml evacuated pyrex bottle, one end of which was fabricated with a thin 1.27 cm O.D. platinum tube and a vent break-off seal. This end seal is broken off in the vent by pulling on a Nichrome wire attached to it. After collection, the Pt tube is cold welded shut with a pair of special pliers.

These gas samples have been analyzed by both gas chromatography and mass spectroscopy and the results, shown in Appendix D, were published by Graeber, et al, in the Journal of Volcanology and Geothermal Research, 1978.

More recently, Dr. Paul Greenland at HVO collected gases from fumaroles and eruptions using both Naughton type tubes and also a pump to suck the gases through titanium tubing into flow through bottles containing sodium hydroxide or ammonia cadmium solutions. His results, reported in the HVO monthly reports are interesting as he was able to analyze the samples practically in the field so there was minimal delay between collection and analysis.

#### Condensates.

Condensates were also collected from fumaroles using a manual peristaltic pump and tygon tubing. These were analyzed at Sandia by Merrill and his results are shown in Appendix C.

#### Corrosion in Fumaroles and Drill Holes.

Early results were included in the June 1976 report. Samples exposed after that period, and which were recovered, were sent to Sandia for analysis. Those results are not known.

Samples were also exposed in the S drill hole in the Kilauea-Iki crater on 10/1/76. The hole was 43.58 m deep and the temperature at the bottom was 887°C. A string containing 8 samples, strung on nichrome wire reinforced with refrasil was hung at 43 m depth. It was not placed on the bottom as the samples would sink in the semi-molten rocks which would solidify around them, thus retrieval would be impossible. Another string was hung at 3 m depth. Mr. R. Okamura from HVO checked the holes a week later (10/8/76) and found that the deep samples were lost: only pieces of very brittle wires were recovered. The shallow samples showed negligible attack.

Samples were also exposed to the fumaroles on the summit of Mauna Loa on 8/20/78. The attacks seen were negligible as the exposure times were only about 1-1/2 hours. Samples could not be left there as climbing to the summit takes at least a day and is also very exhausting.

## Laboratory Experiments.

The results obtained from the field tests are direct but difficult to assess their relevance because of variations in gas compositions and temperatures. Thus, determining the major corrosion mechanisms and sorting out the contribution of the different corrosion species on the overall corrosion kinetics is difficult. Also, field tests set-ups are subject to opportunity, weather, wildlife, and tampering. Thus an artificial fumarole was constructed (June 1976 progress report). In the preliminary tests, Al alloys 2017, 6061-T650, and 7075-T65 were exposed to 3 mole percent SO<sub>2</sub> and 97 mole percent steam at 110°C. The above gas mixture was in the range of gas compositions found by Finlayson, et al (Geophysical Monograph, No. 12, American Geophysical Union) and Heald, et al (Jnl. of Geophysical Research, Vol. 68, No. 2, Jan 1963). The results were discouraging as the corrosive attack was significantly slower than those observed from the field tests. Aluminum and Al alloys are attacked by wet SO<sub>2</sub>. It is known that SO<sub>2</sub> destroys the passivating film and causes pitting. The kinetics of the attack also depends on the condition of the specimen surface. The presence of fine particles such as finely divided charcoal greatly increases the corrosion kinetics on steels, but rust is supposed to decrease the attack. Catalysts such as Pt and iron oxide oxidizes the SO<sub>2</sub> to SO<sub>3</sub> and the surface acid so formed may cause rapid attack. Corrosion rates increased, but was still slower than those found under field conditions when the samples were placed in sulfur beds over which was passed the steam/SO<sub>2</sub> gas mixture. The above was tried because at most fumaroles, especially the No. 6 hole of the 1971 flow,, the entrance and downwind sides were covered with heavy sulfur deposits formed when SO<sub>2</sub> was reduced to elemental S by the H in H<sub>2</sub>S or in steam. The corrosion kinetics were also faster in the No. 6 hole as compared to the No. 7 hole in the same lava flow. The No. 7 fumarole was at one end of a deep ditch, 2-10 m. in depth, thus there was considerable mixing of the atmosphere with the fumarolic gases. Also, there was less sulfur around the hole and the exposed samples are hanging vertically in the gaseous environment unlike the case at the No. 6 hole where the samples are partially embedded in sulfur. At this hole, the vent extended almost horizontally into an embankment, but had several turns. Thus, the samples could be placed only near the vent entrance. Attempts to place the samples deeper in the hole was made impossible by the SO<sub>2</sub> and the steam issuing out of its mouth.

It was obvious from these preliminary tests that the simple SO<sub>2</sub>/steam atmosphere did not simulate the fumarolic conditions satisfactorily. Also, there were problems with the equipment: the silica glass furnace tubes broke frequently, steam condensed in the tubes, steam flow rates were difficult to monitor accurately, SO<sub>2</sub> leaked from the teflon seals and there were strong objections to the pollution caused by the exhaust SO<sub>2</sub>.

A new set-up was constructed using four industrial-type tube furnaces. These allowed accurate temperature control and were also leak-proof. A detailed literature survey, especially reviews of the pertinent thermochemical diagrams were made to help evaluate the possible gas-metal reactions, (see the report submitted in June 1978). The report also included the CO<sub>2</sub>+SO<sub>2</sub>+CO+H<sub>2</sub>O, SO<sub>2</sub>+H<sub>2</sub>S+H<sub>2</sub>O, and SO<sub>2</sub>+O<sub>2</sub>+A gas mixtures which could be formulated to have oxygen and sulfur fugacities similar to those found in volcanic atmospheres. A few trial runs were made using a number of steel samples. These experiments could not be continued for the following reasons.

Sufficient funds was not available to buy a gas chromatograph. Attempt made to have the use of or to borrow one from the other departments prove unsuccessful. A chromatograph was necessary to determine whether the desired gas mixture were obtained. Also, resources were lacking to build a safe venting system for the exhaust gases. The existing vent hood corroded and the presence of SO<sub>2</sub> in the nearby classrooms and laboratories was clearly evident from the smell. This pollution was intolerable. Fortunately, the Mechanical Engineering Department now has a H-P gas chromatograph in the Renewable Energy Laboratory and it is hoped that the planned experiments can be carried out in the near future.

#### Heat-transfer Experiment.

Dr. H. Hardee from Sandia Laboratories brought a stainless steel gage to determine the heat transfer coefficients of lava at the 1977 eruption. The gage was about 50 mm dia and 2 m long and it was necessary to immerse one end of the gage in the lava and measure the changes in temperature with time. It was at first thought that the pole was too short and would require that the researchers approach perilously close to the lava river. So plans were made to acquire pipe sections which could be added to one end of the pole. However, the U.H. group felt that the delay could mean cancellation of the experiment as the eruption could cease at any time. They showed that the heat transfer gage could be used as is by cutting an ohia branch the same length as the pole and simulated the experiment.

The experiment lasted about 5 minutes as the heat became unbearable but mainly because the current in the lava river slowly but inexorably tore the pole from the grasp of the three people holding it and literally swallowed the pole. The eruption ceased the next day at 4:25 p.m. and the lava drained back into the vent. This left a channel about 3 m deep and roughly 25 m wide, and no trace of the pole was found in that channel.

The results of the experiment would be a valuable supplement to the data obtained from heat transfer experiments conducted at Sandia using remelted lava rocks. However, the U.H. group have not received any communication from Sandia regarding the above results.

#### Temperature Measurements.

These were the most successful experiments as they were the simplest. In fact, with the current availability of small, portable direct reading digital thermocouple read-out units, tests will be easier yet. The temperatures in the fumaroles of the 1971 flow in the Halemaumau crater varied from 90°C to 120°C. A couple of attempts were made to get continuous temperature records, e.g. for 2 hours at No. 7 fumarole using a portable electrical generator, and for 15 days at No. 6 fumarole using a battery operated chart recorder. Temperatures varied in a cyclic manner with a high of 115°C and a low of 83°C in the No. 6 fumarole. Minor variations superimposed on the above were probably due to rain and wind gusts.

The temperatures in fissures at a more recent eruption site (1975) was about 240°C. Two 4-in. dia holes were drilled into the solidified lava lake in the Kiluea-Iki crater by Sandia in August and September, 1976. The temperature at the bottom of the S hole was 704°C. The temperatures measured by R. Okamura of HVO in the same hole, Fig. 1, was 887°C at the bottom. The first temperature was measured soon after drilling and was low possibly because of the chilling effect of the mud and water used in the drilling process. The drill hole temperatures were measured again on May 8, 1977, see Fig. 1. Note that the two temperatures shown are from two different holes, the 1976 data is from the S hole and the 1977 data is from the N hole. It was not possible to measure the temperatures in the S hole in 1977 because of an obstruction in the hole at about 4 m. depth.

At the 1977 eruption, the temperatures were about 850°C in hot cracks in recently (1-3 day old) solidified lava flows and about 1050°C-1100°C in the flowing lava river about 150 m. from the eruption. In future projects, attempts should be made to get continuous temperature records at selected sites using either battery operated chart recorders or recorders powered by a portable generator. This capability will assist in getting detailed heat transfer data also.

#### Summary.

Useful information was gained from this project, as reported in this and in previous reports, as well as in several papers published by Sandia personnel. The U.H. group must admit to a certain sense of dissatisfaction mainly because the results obtained were not commensurate with the effort expended. Difficulties were due to lack of sufficient resources to get the equipment needed and also because of inexperience in the field, particularly at the eruption site. However, the necessary dues have been paid for the experience gained. This experience will be helpful, both in planning and in carrying out further work. It is conceivable that geothermal research would again be promoted quite urgently if the events occurring in the Middle East decreased oil supply.

## Appendix A

Summary of results of materials compatibility exposures at 1977 Kilauea eruption.

Approximate severity of oxidation based on visual, macroscopic examination of samples:

- 0 = almost negligible (slight tarnishing or blackening of surface)
- 1 = very slight (generally limited to blackening of surface)
- 2 = noticeable light corrosion (darkening and slight roughening of surface; incipient formation of a scale layer?)
- 3 = more pronounced corrosion (obvious formation of a scale which is beginning to flake off, or significant alteration of the surface)
- 4 = severe corrosion (significant loss of material from the specimen)

	<u>String #1</u> 900° gas vent, 18 hr	<u>String #2</u> 850°C crack in lava, 24 hr	<u>String #3</u> 1085°C lava river 10 min	<u>String #4</u> 850-600°C crack in lava, 6 hr
molybdenum	3	-	-	-
tungsten	3	-	-	3
WC 3015 alloy (Nb-Hf-W-Ti-Zr)	1	-	-	-
nickel	2	-	-	4
iron	3	-	-	3
Fe70-Ni30	3	-	-	3
446 SS	2	-	-	-
302 SS	2	1	2	2
304 SS	-	-	-	2
316 SS	2	2	-	2
A-286 SS	2	2	2	2
RA 330	1	1	2	1
310 SS	0	1	0	2
nichrome	1	-	0	1
Multi-phase 35N alloy (Ni-Co-Cr-Mo)	2	-	1	1
Inconel 600	1	1	1	1
Inconel 718	0	-	-	1
Rene 41	-	-	-	1
Hastelloy C	-	-	1	1
Hastelloy C-276	2	1	0	1

Private Communication: P. Modreski

Appendix B

FUMAROLIC GASES

	<u>1971</u>	<u>1974</u>	<u>1975</u>
date	11/7/75	12/5/75	12/4/75
T°C	122°	94°	237°
N <sub>2</sub>	2.60	.39	3.47
O <sub>2</sub>	.39	.002	.88
Ar	.020	.0019	.016
H <sub>2</sub>	.0003	.0009	.0007
CO <sub>2</sub>	1.81	14.5	.53
CO	.004	.002	.002
SO <sub>2</sub>	.039	.99	.002
SO <sub>3</sub>	.51	.23	.13
HCl	.013	.003	.012
HF	.0003	.0004	.0007
H <sub>2</sub> O	94.6	83.9	95.0

Private Communication: E. Graeber

Appendix C

CHEMICAL ANALYSIS OF CONDENSATES

<u>Sample</u>	<u>pH</u>	<u>ppm</u> <u>SO<sub>2</sub></u>	<u>ppm</u> <u>SO<sub>4</sub><sup>=</sup></u>	<u>ppm</u> <u>Cl<sup>-</sup></u>	<u>ppm</u> <u>F<sup>-</sup></u>
1. Turtoise Dome, 12/4/75, hand pumped	~7	420	<100	4.6	-*
2. Collapsed Turtoise, 12/5/75, pumped, Teflon 12:32 pm	5-6	310	<100	10.3	5.4
3. Turtoise, 12/5/76, hand pumped, 12:45 pm	5-6	460	<100	2.9	6.9
4. 40 min "Solar Collector" with Rock, 12/5/76, 1:10 pm	5-6	-	<100	12.8	0.9
5. '74 Vent hole #1, hand pumped, 12/5/75, 4:15 pm	3-4	>3100	265	8.4	<0.5
6. HVO men's room water faucet	5-6	8.7	<100	5.2	<0.5

\* Insufficient sample for F<sup>-</sup> analysis

Private Communication: P. Modreski



Appendix D

ANALYSES (MOLE %) OF GASES COLLECTED FROM THE  
SEPTEMBER 1977 ERUPTION OF KILAUEA AFTER REMOVAL OF N<sub>2</sub>, O<sub>2</sub> & Ar

<u>Sample No.</u>	<u>H<sub>2</sub>O</u>	<u>H<sub>2</sub></u>	<u>CO<sub>2</sub></u>	<u>CO</u>	<u>SO<sub>2</sub></u>	<u>HCl<sup>+</sup></u>	<u>S/C</u>
1	74.55	5.98	17.30	0.94	0.11	0.96	0.006
5	89.10	ND	5.06	ND	5.45	0.35	1.08
6	96.86	ND	1.99	ND	0.84	0.20	0.42
7	93.58	ND	1.87	ND	4.42	0.12	2.36
10*	64.20	7.09	14.23	8.92	0.46	0.25	0.014
11**	63.19	5.09	30.93	ND	0.46	0.21	0.015

ND: Not detected

+All chloride in condensate reported here as HCl

\*Also contains 2.50% CH<sub>4</sub>, 0.20% C<sub>2</sub>H<sub>6</sub>, 1.43% C<sub>2</sub>H<sub>4</sub>, 0.25% C<sub>3</sub>H<sub>8</sub>,  
0.15% C<sub>3</sub>H<sub>6</sub>, 0.15% C<sub>4</sub>H<sub>10</sub>, 0.10% C<sub>6</sub>H<sub>6</sub>, 0.05% C<sub>6</sub>H<sub>5</sub>CH<sub>3</sub>.

\*\*Also contains 0.18% CH<sub>4</sub>.

Private Communication: E. Graeber

Fig. 1. The S hole temperatures, in the Kilauea Iki drill holes, were measured by R. Okamura of HVO on 10/14/76. The N hole temperatures were measured on 8/5/77. The down temperatures were determined as the T.C. was lowered, and the up data as the T.C. was raised after reaching bottom.

