

# Oxygen and Sulphur Fugacities of Magmatic Gases Directly Measured in Active Vents of Mount Etna [and Discussion]

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## Oxygen and sulphur fugacities of magmatic gases directly measured in active vents of Mount Etna

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Solid-electrolyte sensors were used to measure the oxygen and sulphur fugacities of magmatic gases in vents located at an elevation of about 3170 m on the northeast flank of the NE crater of Mt Etna in July 1970. The temperature of the gas ranged from 773 to 1057 °C. The least-squares fit of 13 measured  $\lg f_{\text{O}_2}$  values yielded the relation:

$$\lg f_{\text{O}_2} \text{ (atm)} = 2.175 - 15110/T \text{ (K)},$$

which gives values of 0.9 to 2.2  $\lg f_{\text{O}_2}$  units higher than those obtained in the Makaopuhi lava lake, Hawaii, within the temperature range measured, the difference increasing with decreasing temperature. Laboratory study of a water-quenched sample of the Etna lava and calculations of gas equilibria indicate that the difference is due partly to the intrinsically higher  $f_{\text{O}_2}$  of the Etna magma and partly to the mixing of air with the Etna gases. The measured  $f_{\text{S}_2}$  value was  $10^{-2.4}$  atm at 860 °C.

### 1. INTRODUCTION

The field work reported in this paper was carried out in July 1970, when the authors joined the international scientific expedition on Mt Etna organized by Professor H. Tazieff. The initial plan was to determine the oxygen fugacity values of the magmatic gases emitted from Bocca Nuovo, which started out as a small but very active gas vent and later collapsed to form a deep fuming pit in early 1970. The field measurements were made, for this reason, in small horn-shaped gas vents (commonly referred to as hornitos) at the NE flank of the NE crater, using a solid-electrolyte oxygen sensor probe. Sulphur fugacity values were also measured in one of the vents using a new solid-electrolyte sensor developed for such a use. A small batch of the lava was scooped from a nearby lava well and quenched in water. The oxygen fugacity values of this sample were determined later in our laboratory to gain an insight into the behaviour of the gases in the vents relative to that of the lava.

### 2. OXYGEN FUGACITIES OF THE MAGMATIC GASES

#### (a) *Sites of measurements*

The small gas vents (hornitos) were located at the NE flank of the NE crater at an elevation of about 3170 m, the approximate location of which is shown in figure 1. A cluster of several vents of variable size trend northwestward at irregular intervals (10 to 50 m). At the NW extension and downhill of the cluster, lava was constantly circulating in a well and farther downhill it was flowing out of fissures. The measurements were made in two medium-sized (about 30 cm across) vents on 10 and 12 July. The lava level rose on 12 July probably because of temporary build-up of gas pressure in this particular portion of the subterranean plumbing system of the summit region. Rapidly circulating incandescent lava gradually rose to the orifices of the vents till minor amounts were spewed out by the high-velocity gas stream. The process

started from the vent at the highest elevation and shifted in sequence northwestward to vents at lower elevations. The  $f_{O_2}$  measurement was terminated when the rising lava touched the probe and damaged it.

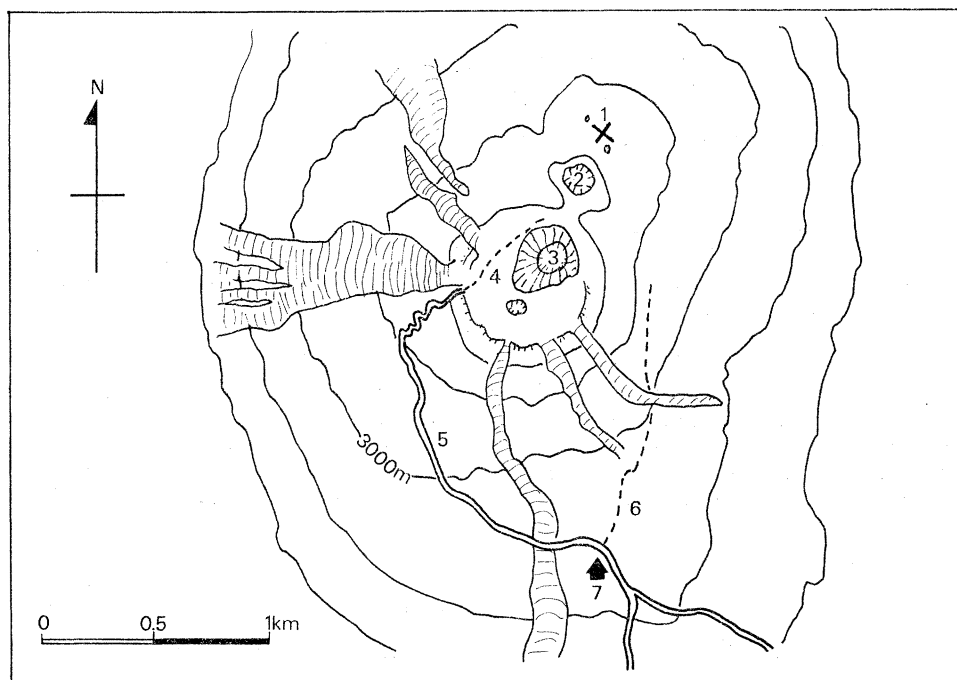


FIGURE 1. A map showing the approximate location of the gas vents (x). Contour interval 100 m; datum is mean sea level. (1) Vents where measurements were made; (2) NE crater (Bocca N.E.); (3) central crater (Cratere Centrale); (4) Bocca Nuovo; (5) jeep road; (6) trail; (7) old observatory.

### (b) Method

A solid-electrolyte  $f_{O_2}$  sensor similar, in principle, to the one used by Sato & Wright (1966) in the Makaopuhi lava lake, Hawaii, was used in combination with a sheathed chromel-alumel thermocouple. The design of the sensor probe is schematically shown in figure 2. It differs from the earlier probe in that air flow is used as the internal  $f_{O_2}$  reference instead of the Ni-NiO buffer. The air reference type is superior to the solid-buffer reference type in applications where the change of temperature is rapid and also where the temperature is below about 600 °C, because of much more rapid temperature response. It suffers from the fact that barometric corrections are necessary where the altitude is high, however. The effects of variation in humidity and the presence of minor amounts of volcanic gases can be ignored so long as the air is pumped from a location where a man can breathe comfortably. A total of 4% of  $H_2O$ ,  $CO_2$ ,  $SO_2$ , and other gases in air decreases the  $f_{O_2}$  reading of the sensor by less than 0.02 lg unit; which is negligible compared to other sources of error such as temperature and barometric pressure variations. The solid electrolyte used is 9 mol % yttria-stabilized zirconia instead of more commonly used 15 mol % calcia-stabilized zirconia. The yttria-stabilized zirconia has a higher ionic conductivity, particularly at low temperatures (see Sato 1971).

The solid-electrolyte probe was tied together with the sheathed thermocouple side by side and the whole assemblage was lowered slowly into the gas vent. The reference air was pumped into the interior bottom of the electrolyte tube at a rate of about 40 cm<sup>3</sup>/min by means of a

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rubber-bulb pump with a pressure reservoir. The flow was fed through a long Tygon tube to the top of the probe assemblage, a stainless-steel capillary tube that runs through the centre of the probe handle and then a small hole in a 4-hole alumina insulation tube placed inside the electrolyte tube. The return air flow escaped to open atmosphere through another hole in the alumina tube and up through the interior of the probe handle without contaminating the

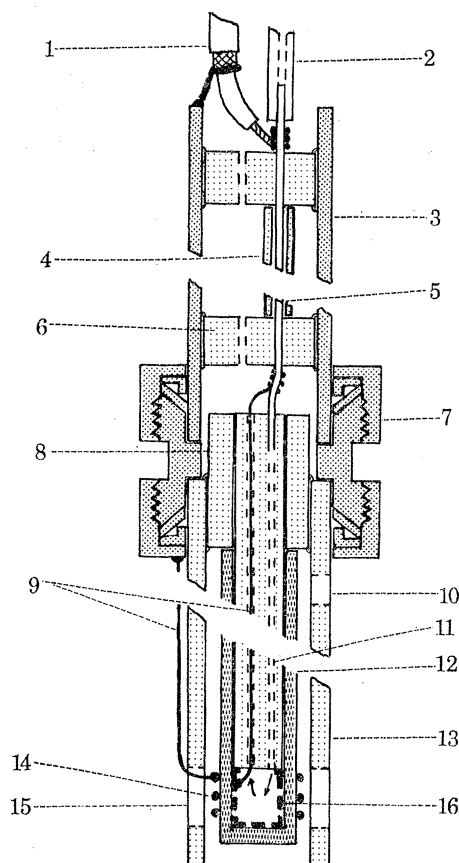


FIGURE 2. The design of the oxygen fugacity sensor probe used in this study. 1, Teflon-insulated shielded cable; 2, Tygon tubing for reference gas supply; 3, no. 316 stainless-steel pipe (3 m long, 12.7 mm o.d.); 4, mullite tubes for insulation; 5, no. 316 stainless-steel capillary tubing (1.6 mm o.d., 1.2 mm i.d.); 6, 2-hole mullite insulator; 7, stainless-steel connexion; 8, alumina tube collar; 9, platinum wires; 10, gas venting hole; 11, 4-hole (can be 2-hole) alumina tube; 12, 9 mol % yttria-stabilized zirconia electrolyte tube (15 cm long and 9.5 mm o.d.); 13, alumina protection tube; 14, platinum coil; 15, window for gas; 16, platinum basket.

magmatic gas. The e.m.f. across the wall of the electrolyte tube was transmitted first through two platinum wires, then through the stainless-steel pipe (positive circuit) and capillary tube (negative circuit), and finally through a Teflon-insulated shielded cable. The electrometer used was a battery-operated, high-input resistance ( $10^{14} \Omega$ ) type. The outputs of the electrometer and the cold-junction compensator of the thermocouple were connected to a 4-channel strip-chart recorder and also to a cassette analogue magnetic tape recorder, both battery-operated. The e.m.f. of the  $f_{O_2}$  probe and of the thermocouple were recorded as a function of time and the depth from the orifice of a vent.

## (c) Results

The results obtained are listed in table 1. The vent identification codes EH-1 and EH-2 are simply for convenience, and do not carry geological significance. The depth was measured from the orifice of a vent. The  $\lg f_{O_2}$  values were computed by the formula (see Sato 1971)

$$\lg f_{O_2} = \frac{20.16 \times E \text{ (in mV)}}{t(^{\circ}\text{C}) + 273} - 0.845, \quad (1)$$

where  $E$  is the e.m.f. of the exterior (magmatic gas) side of the solid-electrolyte cell measured in reference to the interior (air) side and expressed in millivolts, and the constant 0.845 is the logarithm of the oxygen fugacity of air at the elevation of 3170 m above sea level.

TABLE 1. TEMPERATURE AND OXYGEN FUGACITY VALUES OF ETNA GASES

vent and date	depth/cm	temp./ $^{\circ}\text{C}$	sensor e.m.f./mV	$\lg f_{O_2}\dagger$
EH-1, 10 July 1970	0	1018	-570	-9.74
	10	1031	-560	-9.50
	20	1044	-540	-9.11
	30	1057	-545	-9.10
EH-2, 10 July 1970	0	773	-580	-12.02
	5	916	-560	-10.34
	20	1023	-542	-9.28
	50	1044	-530	-8.96
EH-2, 12 July 1970	20	967	-560	-9.95
	10	941	-570	-10.31
	5	911	-600	-11.06
	10	941	-580	-10.48
	20	992	-578	-10.06
	30 (?)	1069	‡	

† Values used for the calculation of equation (2). Accuracy is perhaps about 0.2 lg unit because of minor fluctuations in the e.m.f. readings.

‡ No reading because lava erupted and damaged the sensor.

The measured  $\lg f_{O_2}$  values of the gases in the vents are plotted against the reciprocal absolute temperature in figure 3. Although there is some scatter, the data show a fairly good linear correlation. The least-squares fit of the data yielded the relation:

$$\lg f_{O_2} \text{ (atm)} = 2.18 - 1.51 \times 10^4/T. \quad (2)$$

The solid line in figure 3 expresses this equation. The significance of this equation is open to discussion. It was observed at one time during the measurements that when a strong gust of wind blew, the  $\lg f_{O_2}$  value became more positive by as much as 1.5 units, indicating that the magmatic gas in the vent was subject to contamination by air, the degree of which depended on the wind velocity. The condition of the measurements was not as good as that which normally prevails in a drill hole in a quiet lava lake. It could well be that the lower margin of the shaded area in figure 3 represents the  $\lg f_{O_2}$  values of the least contaminated magmatic gas of Etna. The equation, nevertheless, serves the purpose of characterizing the average  $\lg f_{O_2}$  values of the gases in these vents. For comparison, the equation characterizing the measurements made in drill holes in the Makaopuhi lava lake before the appearance of a high  $f_{O_2}$  peak (Sato & Wright 1966) is also plotted in figure 3. It can be seen that the Etna values are higher than the Hawaiian values by 0.9 to 2.2 lg  $f_{O_2}$  units within the temperature range measured, the difference increasing with decreasing temperature.

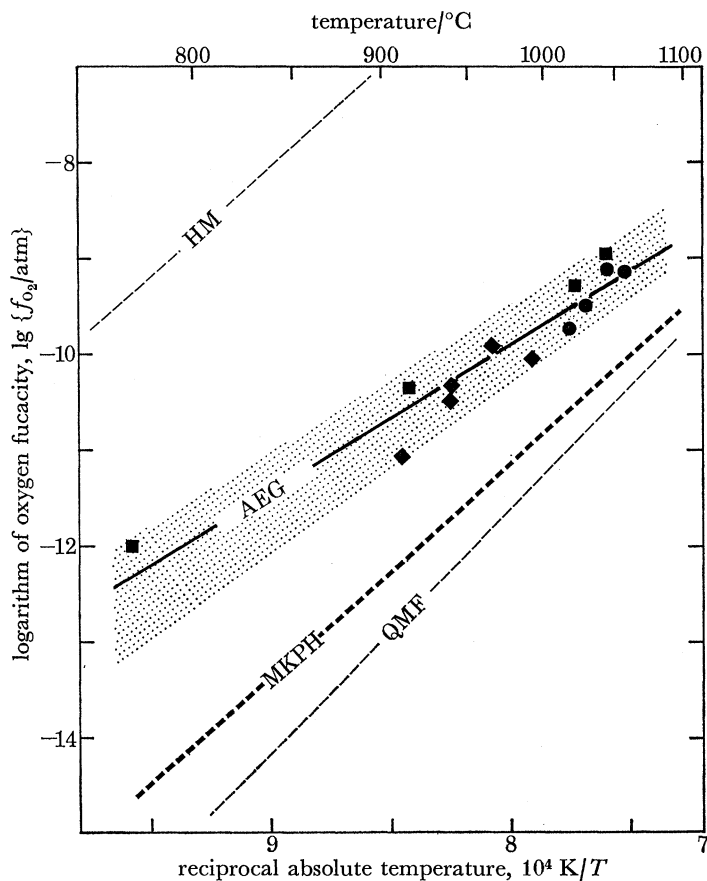


FIGURE 3. A  $\lg f_{O_2}$  against temperature plot of values obtained in the gas vents of Mt Etna. The solid line (AEG) represents the least-squares average of the 13 measured values of the Etna gases. The shaded area indicates the approximate range of the measured values. The dashed line (MKPH) represents the average value obtained in drill holes in Makaopuhi lava lake, Hawaii (Sato & Wright 1966). The dashed lines (HM and QMF) are the equilibrium values for the hematite-magnetite and the quartz-magnetite-fayalite buffers, respectively. Symbols: ●, EH-1 series; ■, EH-2, 10 July 1970 series; ◆, EH-2, 12 July 1970 series; listed in table 1.

### 3. OXYGEN FUGACITIES OF THE LAVA

#### (a) *Lava sample*

A small quantity of lava circulating in a lava well about 50 m NW of the vents was scooped up with a long steel bar and immediately quenched in a bucket of water. The central part of the quenched lava least affected by the reaction with air and water was chipped off for the laboratory determination of the  $f_{O_2}$  values. The temperature of the lava measured at the time of sampling was 1044 °C.

Microscopic examination of the quenched lava sample reveals that the lava was about 50 % crystallized at this temperature. Phenocrysts included strongly zoned plagioclase (30 %), olivine (10 %), clinopyroxene (10 %), and magnetite (2 %). Vesicularity was about 25 %.

#### (b) *Method*

A solid-electrolyte method called the opposing double-tube method incorporating the  $f_{O_2}$  negative feedback principle (Sato 1971, 1972) was used to determine the  $f_{O_2}$  values of about 200 mg of the bulk lava sample. This method has been refined to the extent that the intrinsic



$f_{O_2}$  values of most iron-bearing minerals and also the bulk  $f_{O_2}$  of lunar basalts ( $Fe_2O_3$  0.00 mass %) can be determined in 100 mg quantity with a reproducibility better than 0.2 lg  $f_{O_2}$  unit. A small crucible made of the  $Ag_{60}Pd_{40}$  alloy (Muan 1963) capped with a foil of the same alloy was used as the crucible. The vertical arrangement of the entire assembly differs from that described in Sato (1972), having the reference electrode tube at the top and the sample-electrode tube at the bottom. The crucible was supported by a Vycor capillary rod within the sample electrode tube.

(c) Results

The lg  $f_{O_2}$  values of the quenched Etna lava are plotted against the reciprocal absolute temperature in figure 4. The trace consists of four linear segments presumably representing the above-liquidus ( $> 1140$  °C), ferrous silicate crystallization (1140 to 1090 °C), magnetite

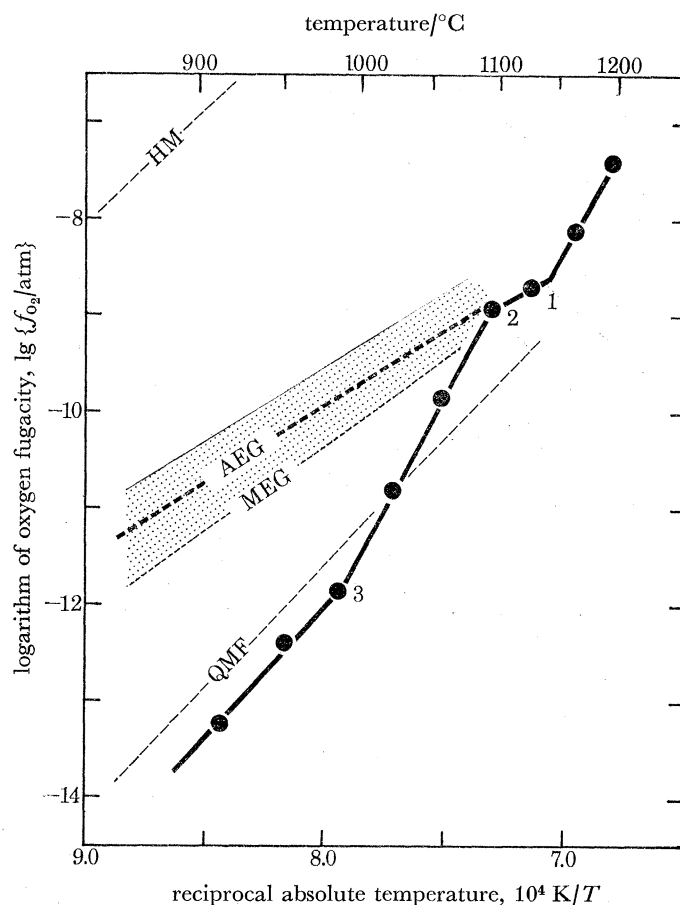


FIGURE 4. The lg  $f_{O_2}$  against temperature plot of a water-quenched Etna lava obtained near the gas vents. The changes in slope are presumably due to (1) the start of crystallization of ferrous silicates (olivine and clinopyroxene), (2) that of magnetite, and (3) disappearance of liquid (solidus), as marked in the figure. The lines marked AEG and MEG represent the average and the minimum Etna gas values, respectively. The dashed line marked HM and QMF represent the hematite-magnetite and the quartz-magnetite-fayalite buffer values, respectively.

crystallization (1090 to 985 °C), and subsolidus ( $< 985$  °C) stages. The phase relations study of this particular lava has not been made by the authors, and the above interpretation is based on experience with other basalts (Sato 1968). The phase relations study of the 1971 Etna lava under controlled  $f_{O_2}$  ( $10^{-5}$  atm at 1200 °C) by M. J. Downes (1973, this volume) is

essentially in agreement with the above interpretation except that plagioclase appeared as the liquidus phase at about 1200 °C. The crystallization of plagioclase, which contains no substantial amount of iron, does not significantly affect the  $f_{\text{O}_2}$  value of the melt, which in normal terrestrial basalts depends primarily on the ferric to ferrous ratio. The  $f_{\text{O}_2}$  value of this water-quenched Etna lava as determined by the solid electrolyte method is about  $10^{-7.3}$  atm at 1200 °C.

#### 4. SULPHUR FUGACITIES OF THE MAGMATIC GAS

##### (a) Site and method

The measurements were made in the same vents as those used for  $f_{\text{O}_2}$  measurements. As the solid-electrolyte sulphur sensor described below cannot be used reliably above about 840 °C and the gases in the vents were hotter than this limiting temperature (see table 1), only a few measurements were made near the orifice of the vents, where temperature variation was severe and air contamination was apparent.

The solid-electrolyte  $f_{\text{S}_2}$  sensor used consists of an exterior film of  $\text{Ag}_2\text{S}$ , Ag  $\beta$ -alumina electrolyte, and Ag internal reference as described by Sato (1971). It is essentially a silver concentration cell, in which the activity of silver in the  $\text{Ag}_2\text{S}$  film varies within a range depending on the sulphur fugacity of the gas it is exposed to. The  $f_{\text{S}_2}$  value is obtained by the equation

$$\lg f_{\text{S}_2} = \frac{20.155 \times E \text{ (in mV)} - 9175}{t(^{\circ}\text{C}) + 273} + 3.61 \quad (3)$$

above 587 °C and below the melting-point of  $\text{Ag}_2\text{S}$  which is about 840 °C (Hansen 1958).

##### (b) Results

The results obtained were not up to expectation. As mentioned before, the conditions encountered in the vents were not favourable for good  $f_{\text{S}_2}$  measurements. Within the vents where the gases are less contaminated and uniform, the temperature was too high for the sensor. Outside of the vents where the temperature was low enough, the gases were oxidized and diluted by mixing with air and the sensor registered 12 to 15 mV at most. This does not mean that sulphur was absent, however. Most sulphur existed as  $\text{SO}_2$ , and by virtue of the relations:

$$\text{S}_2 + 2\text{O}_2 = 2\text{SO}_2, \quad K = f_{\text{SO}_2}^2 / f_{\text{S}_2} f_{\text{O}_2}^2, \quad (4)$$

$$\lg f_{\text{S}_2} = 2 \lg f_{\text{SO}_2} - 2 \lg f_{\text{O}_2} - \lg K, \quad (5)$$

the quantity  $\lg f_{\text{S}_2}$ , to which the sensor responds, becomes very small when  $f_{\text{O}_2}$  is large relative to  $f_{\text{SO}_2}$ .

One measurement made at a depth of 5 cm from the orifice of vent EH-2 produced a reasonably steady reading of 120 mV at 860 °C. This temperature exceeds the melting-point of  $\text{Ag}_2\text{S}$  and the sulphide must have existed as a liquid film at the surface of the electrolyte. As the temperature is only slightly higher than the melting-point, it was assumed that the error introduced by the solid to liquid transition is small and that equation (3) could be used as an approximation. The  $f_{\text{S}_2}$  value thus computed is  $10^{-2.4}$  atm at 860 °C. The accuracy of this value, however, is presumably low (perhaps  $\lg f_{\text{S}_2} = -2.4 \pm 0.5$ ) for the reason discussed above.



## 5. DISCUSSION OF THE RESULTS

The discussion presented here mainly concerns the pattern of the oxygen fugacity of the magmatic gas in relation to that of the water-quenched lava. As seen in figure 4, the average Etna gas values intersect the lava values at about 1090 °C. When a magmatic gas mixture is released from a lava, the  $f_{O_2}$  values should be identical in the two phases at the temperature of the release. The temperature of the circulating lava at the bottom of the vents was estimated

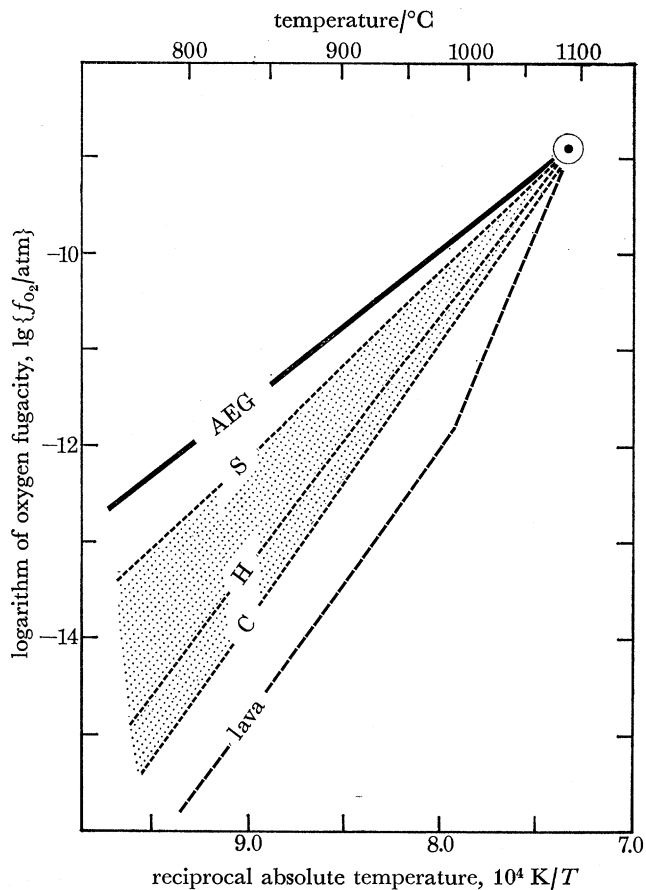


FIGURE 5. Comparison of the slope of the average Etna gas values (AEG) with those of gases of constant total composition on a  $\lg f_{O_2}$  against  $1/T$  plot to examine the degree of air contamination. The dashed lines marked S, H and C represent gases consisting entirely of  $SO_2-S_2$ ,  $H_2O-H_2$ , and  $CO_2-CO$ , respectively. The shaded triangular area indicates the range of the slope of a gas consisting of C, H, S, and O. The broken line (lava) shows the slope of the lava. These lines all originate at  $\lg f_{O_2} = -8.9$  and 1090 °C.

at about 1070 °C from the highest gas temperature measured (table 1). The discrepancy of 20 °C is reduced by more than 10 °C if we obtain the intersection temperature with the lower margin of the  $f_{O_2}$  distribution zone by assuming that the gas was more or less contaminated by air (combustion) and that the lowest values obtained are closest to the values of uncontaminated magmatic gas. The observed rise of  $f_{O_2}$  with increasing surface wind velocity described earlier appears to strongly support this assumption. The remaining difference of less than 10 °C could be resolved if it is assumed that all the gases measured were contaminated – an indeed unpleasing assumption. Alternatively, the difference could be explained by assuming that the gases were released at around the intersection temperature at a certain depth

where the lava temperature was higher and quickly rose to the surface without re-equilibrating with the lava. The vesicular nature of near-surface lava columns in active volcanoes strongly suggests that gases are released (probably continuously) over a range of depth and temperature as the load pressure decreases and the crystallization proceeds. The observation by Moore (1965) that the vesicularity of submarine pillow lavas increased as an inverse function of the depth of extrusion also supports this interpretation. The rapid equilibration between the gas and the lava observed in drill holes in the Makaopuhi lava lake (Sato & Wright 1966), however, tends to negate the assumption that the gases escaped without re-equilibrating with the lava.

An independent method of checking the degree of contamination is to compare the slope of the observed  $\lg f_{O_2}$  against  $1/T$  plot with those of gases of fixed total atomic composition obtained by equilibrium computation similar to those made by Heald, Naughton & Barnes (1963). It is necessary to know the composition of the gas at the point of release from lava to do this accurately, however. This difficulty can be circumvented by assuming that the gas mixture is mainly composed of  $H_2O$ ,  $CO_2$ , and  $SO_2$  and minor amounts of  $H_2$ ,  $CO$ ,  $H_2S$ , and  $S_2$  in the proportions compatible with the  $\lg f_{O_2}$  value at the intersection temperature. Extreme cases are gases composed entirely of  $H_2O-H_2$ ,  $CO_2-CO$ , or  $SO_2-S_2$ . These extreme cases are shown in figure 5, all originating at  $\lg f_{O_2} = -8.9$  and  $1090^\circ C$ .

The slope of a mixture of an intermediate composition (e.g. 45%  $H_2O$ , 30%  $CO_2$ , 15%  $SO_2$ , 10% minor constituents) will fall within the range covered by the three extreme cases (shaded triangular area of figure 5). As readily recognizable in figure 5, the Etna gas has a gentler slope than any of the extreme cases. This behaviour is explainable only by increasing addition of oxygen with decreasing temperature.

According to Fudali (1965), and the data obtained by the oxygen cell method (M. Sato, unpublished data), Kilauea lavas have  $f_{O_2}$  values lower than  $10^{-8}$  atm ( $10^{-3}$  Pa) at  $1200^\circ C$ . This is more than 0.7 lg unit lower than the Etna lava value. The slope of the  $\lg f_{O_2}$  against  $1/T$  plot of the Makaopuhi gas data is steeper than that of the Etna gas as evident in figure 3. From the discussions presented above, it is concluded that the differences in magnitude and slope of the  $\lg f_{O_2} - 1/T$  plot between the Etna gas and the Makaopuhi gas are mainly due to both the higher  $f_{O_2}$  of the parental lava and the higher degree of air contamination for the Etna gas.

The authors are grateful to Professor H. Tazieff for soliciting the participation and providing various services and supplies in the international expedition to Etna. Priestley Toulmin, III and Edwin Roedder kindly reviewed the manuscript. We also thank Dallas Peck for encouraging our participation in the expedition. Publication is authorized by the Director, U.S. Geological Survey.

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### Discussion

MR A. T. HUNTINGDON: Do the gases buffer the lava or does the lava buffer the gases?

DR SATO: This is a case of action–reaction and, strictly speaking, both will be affected as the result of the interaction. In terms of buffering capacity for comparable volumes, however, the lava is predominantly in control of the oxygen fugacity. To illustrate, suppose we have a lava containing 4 mass %  $\text{Fe}_2\text{O}_3$  and 8 mass %  $\text{FeO}$ , and in contact with the lava a gas mixture containing 2 vol. % excess  $\text{H}_2$  (the amount in excess of what is required of in order to have the same  $f_{\text{O}_2}$  as the lava before the interaction) at 1000 °C. Calculations show that to reduce 1 mass % of  $\text{Fe}_2\text{O}_3$  to  $\text{FeO}$ , about 600 times as much (in volume) gas would have to react with the lava, and this would result in the decrease of the  $f_{\text{O}_2}$  of the lava by less than 0.7 lg unit, while the  $f_{\text{O}_2}$  of the gas would increase by as much as 4 lg units. This kind of deduction applies, however, only after the release of the gas phase from the lava. A much more complex and different situation exists at the time of the release of the volatiles from the lava. Loss of hydrogen as  $\text{H}_2$  from the hydroxyl radicals in a silicate melt, for example, tends to raise the  $f_{\text{O}_2}$  of the lava substantially. Loss of sulphur as  $\text{SO}_2$  or  $\text{S}_2$  from a melt in which sulphur is dissolved as  $\text{S}^{2-}$  ion, on the other hand, tends to lower the  $f_{\text{O}_2}$  of the lava equally substantially. The effect of the loss of volatiles on the magmatic  $f_{\text{O}_2}$  is currently under investigation.

DR M. S. BECK: At Bradford University we intend to develop a dynamic model which will simulate the time varying behaviour of Mt Etna on either an analogue or digital computer.

In order to prepare a dynamic model it is necessary to have data measured either continuously or at frequent intervals and the data should be accurately timed.

Since dynamic model building could form a useful future technique, it would be most helpful if all research workers could try to ensure that their data are collected in a satisfactory form for this purpose. In particular I would like to ask Dr Sato whether he has been able to collect data in the form covered by the requirements given below.

I believe that the following steps could help to ensure that data is in a suitable form:

- (1) All measurements should be accurately timed to the nearest second. A good watch which can be checked against radio time signals should be satisfactory for this purpose.
- (2) Continuous records of data on a chart recorder, tape recorder, etc. are ideal. If continuous recording is not possible and discrete measurements must be taken, then these measurements should be taken sufficiently frequently to follow the pattern of any changes.
- (3) Records should be carefully retained for future use. For dynamic model building the actual measured values are all required. If readings are averaged over a period of time, these averages may not be suitable for dynamic model building.

We would be particularly pleased to hear from any research workers who are in a position to gather data suitable for dynamic model building and we will be pleased to discuss suitable methods of recording the data (please contact Dr M. S. Beck, School of Control Engineering, University of Bradford, Great Horton Road, Bradford, Yorkshire, BD7 1DP, England).