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W. L. Fielder, C. A. Stearns,
and F. J. Kohl
Lewis Research Center
Cleveland, Ohio

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REACTIONS OF NaCl WITH GASEOUS SO_3, SO_2, AND O_2

W. L. Fielder, C. A. Stearns, and F. J. Kohl
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

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ABSTRACT

Hot corrosion of gas turbine engine components involves deposits of Na_2SO_4 which are produced by reactions between NaCl and oxides of sulfur. For the present investigation, NaCl single crystals were exposed at 100° to 850°C to gaseous mixtures of SO_3, SO_2, and O_2. The products formed during this exposure depend, primarily, on the temperatures. The four product films were: $NaCl \cdot SO_3$; $Na_2S_2O_7$; Na_2SO_4; and $NaCl$-Na_2SO_4. The kinetics of the reactions were measured.

INTRODUCTION

Hot corrosion degradation of components of gas turbine engines burning relatively clean fuels usually involves deposits of only Na_2SO_4. Two processes have been proposed to account for the deposits: (1) deposition by impaction of NaCl particles on the turbine surfaces and subsequent reaction of the condensed NaCl with oxides of sulfur; and (2) reaction in the hot combustion stream to form Na_2SO_4 with subsequent deposition. Some previous investigators have observed the conversion of NaCl to Na_2SO_4 under real or simulated combustion atmospheres in the presence of oxide scales on turbine-type substrates (1, 2, 3). For example, substrates such as superalloys or Al_2O_3 were exposed to sea salt sprays (leading to NaCl of small particle sizes) and oxides of sulfur at about 650° to 850°C (1). Analyses of certain portions of the substrate surfaces (i.e., by EDS and SEM techniques) suggested that the rates of conversion of NaCl to Na_2SO_4

were moderately rapid. Such experiments, while verifying that conversion of NaCl to Na_2SO_4 does occur, did not clearly differentiate between processes 1 and 2. Furthermore, the influence of the substrates, used in these experiments, upon the rates of conversion was still unclear.

The present study was concerned primarily with observing the surface reactions occuring when solid NaCl is exposed to oxides of sulfur (i.e., the reaction in process 1 that might occur after NaCl impaction) over a broad temperature range without the influence of oxide scales or turbine-type substrates. Therefore, NaCl, either as single crystal or in a 1:1 powder mixture of Na_2SO_4 and NaCl, were exposed to a gaseous mixture of SO_3 in SO_2-O_2 between 100° and 850°C. The products and kinetics of the reactions were determined.

EXPERIMENTAL

Anhydrous NaCl single crystal samples were obtained by cleaving from a high purity boule grown from the melt. For a few runs, samples of anhydrous powder mixtures of 1:1 molar NaCl and Na_2SO_4 were prepared by melting high purity NaCl and Na_2SO_4 at 700°C. Anhydrous, high purity mixtures of SO_2 in O_2 were obtained commercially or were prepared in-house by pressure dilutions from commercial SO_2 and O_2.

A schematic of the thermogravimetric apparatus is shown in Fig. 1. The sample was suspended (inside a quartz tube) in the center of the top furnace. A Pt catalyst, placed in the center of the bottom furnace, was used to produce the desired concentration of SO_3 in the particular SO_2-O_2 mixture flowing through the quartz tube. The system was pretreated by heating the sample and the catalyst to about 600°C under an O_2 atmosphere. The catalyst was then cooled to about 500°C and the sample was brought to the desired temperature. The sample was exposed to the SO_2-O_2 mixture and the weight changes were followed with time using a sensitive electrobalance. Usually, the flow rates of the mixtures, which were controlled by means of mass flow controllers, were about 17 $cm^3\ sec^{-1}$ (0.84 $cm\ sec^{-1}$).

For the anhydrous runs, the H_2O contents in the gaseous atmospheres were less than 20 to 40 ppm as indicated by hydrometric analyses. For a few runs, the influence of H_2O was observed by introducing it into the system by dilution by passing a parallel O_2 flow through an H_2O bubbler. For these runs, the H_2O content was determined with sufficient accuracy using vapor pressure data and measured flow rates.

High pressure mass spectrometric sampling techniques were used to analyze the reactant compositions and to identify the gaseous products. The apparatus and techniques have been discussed previously (4). This technique determines the concentrations of the gaseous reactants and/or products being produced for the actual experimental conditions because it effectively "freezes" the chemical composition of the gas flow. For the SO_2 calibration curves, mixtures of SO_2 and O_2 of known compositions were used. For the SO_3 calibrations, a commercial SO_3 analyzer was used initially to determine the absolute SO_3 concentration produced in a mixture of SO_2-O_2 flowing over the Pt catalyst. At the same time, the mass spectrometer calibration curve was obtained using these SO_3 results and recorded mass peak intensities for SO_3, SO_2, SO, and O_2.

RESULTS AND DISCUSSION

The product films formed on the NaCl surface, when it is exposed to oxides of sulfur, depend primarily upon the temperature of exposure. The four reactions leading to these films are as follows:

$$NaCl(s) + SO_3(g) \rightarrow NaCl \cdot SO_3(s) \qquad [1]$$

$$2NaCl(s) + 2SO_3(g) + \tfrac{1}{2}O_2(g) \rightarrow Na_2S_2O_7(l) + Cl_2(g) \qquad [2]$$

$$2NaCl(s) + SO_3(g) + \tfrac{1}{2}O_2(g) \rightarrow Na_2SO_4(s) + Cl_2(g) \qquad [3A]$$

$$2NaCl(s) + SO_3(g) + H_2O(g) \rightarrow Na_2SO_4(s) + 2HCl(g) \qquad [3B]$$

$$XNaCl\text{-}YNa_2SO_4(l) + SO_3(g) \rightarrow (\tfrac{X}{2} + Y)Na_2SO_4 + Cl_2(g) \qquad [4]$$

Formation of Solid $NaCl \cdot SO_3$

A semi-porous solid film of $NaCl \cdot SO_3$ forms when NaCl at 100° to 150°C is exposed to SO_3, SO_2, and O_2. The film formation process is characterized by reaction [1]. This reaction has been previously used as an analytical method for determining SO_3 concentrations in the presence of SO_2 and O_2 (5). At higher temperatures (still below 400°C), solid $Na_2S_2O_7$ also forms and at increasing concentrations relative to the $NaCl \cdot SO_3$. For example, the solid film produced at 250°C was a mixture containing $NaCl \cdot SO_3$ and $Na_2S_2O_7$. For this analysis, the total $NaCl \cdot SO_3$ and $Na_2S_2O_7$ was determined by sulfate analysis while the $NaCl \cdot SO_3$ alone was determined by titration with base.

The rate of formation of the solid product film of $NaCl \cdot SO_3$ at 150°C was obtained by exposing a NaCl sample to a SO_3-SO_2-O_2 mixture containing 0.1 mole percent SO_3. The weight of the sample initially increased linearly with time (at about 2.7×10^{-4} mg cm^{-2} sec^{-1}). After a relatively short time at this linear rate, the sample weight increased parabolically as shown in Fig. 2. The parabolic rate constant ($(\Delta W/A)^2/t$) was about 2.4×10^{-4} mg^2 cm^{-4} sec^{-1} (i.e., 2.4×10^{-10} g^2 cm^{-4} sec^{-1}). A value for the diffusion coefficent D can be calculated from the parabolic rate constant using the following relation (6):

$$(\Delta W/A)^2/t = 2DCd \qquad [5]$$

where d = the density of the film (g cm^{-3}); and where C = the concentration of SO_3 in the gas (i.e., 3.6×10^{-6} g cm^{-3}). For this calculation, the concentration of SO_3 at the film-substrate interface is assumed to be neglible and the density of the film is estimated to be 2 g cm^{-3}. The calculated value of D is about 2×10^{-5} cm^2 sec^{-1}.

Even though the calculated diffusion coefficient value is similar to those usually obtained for diffusion though liquids (6) (i.e., about 10^{-4} cm^2 sec^{-1}), diffusion through a liquid film does not seem likely as the rate controlling process because no evidence of a liquid film was observed on the NaCl surface at 150°C. A solid-state diffusion process also does not seem likely because it should be characterized by smaller diffusion coefficients. Therefore, it is proposed that the $NaCl \cdot SO_3$ film formation occurs, primarily, by transport of gaseous SO_3 to the surface through rather tortuous paths in the semi-porous solid film which is increasing in thickness with time.

Formation of Molten $Na_2S_2O_7$

Upon exposure of NaCl, at temperatures above 401°C, to SO_3, SO_2, and O_2, molten $Na_2S_2O_7$ is the principal film product. For example, a mass balance calculation showed that the film produced at 420°C, after about 10 min, was essentially molten $Na_2S_2O_7$ with only a slight excess of SO_3. The sample, after exposure for 1.5 hr at 420°C, was quenched to minimize any further change in the film composition. X-ray analysis of a small portion of the tightly adhering film showed only $Na_2S_2O_7$, Na_2SO_4, and the NaCl substrate. After reheating this film at about 500°C in flowing O_2 for about 12 hr, the $Na_2S_2O_7$ was converted to a porous non-adhering film of Na_2SO_4 as indicated by X-ray analysis.

The rate of formation of the $Na_2S_2O_7$ and Na_2SO_4 film was determined at 415°C by exposing a NaCl single crystal to an anhydrous reactant of 0.1 mole percent SO_3 in SO_2-O_2. As shown in Fig. 3, the rate of weight change was parabolic with a parabolic rate constant of 5.8×10^{-3} mg^2 cm^{-4} sec^{-1} from which a diffusion coefficient of about 4×10^{-4} cm^2 sec^{-1} was calculated. This value is in reasonable agreement with those values usually obtained for diffusion processes in liquids and in particular with the value of about 2×10^{-4} cm^2 sec^{-1} reported for the diffusion of $S_2O_7^{-2}$ ions in molten Na_2SO_4 (7) and for SO_3 in a chloride melt (8). Therefore, it is proposed that, for the temperature range of 401° to about 450°C, molten $Na_2S_2O_7$ (reaction [2]) is the principal film product that is formed when NaCl is exposed to the oxides of sulfur. The process becomes diffusion limited with SO_3 and/or $S_2O_7^{-2}$ as the primary diffusing species.

The exact mechanism for the $Na_2S_2O_7$ film formation is still unclear. However, it seems reasonable to propose that, initially, a very thin film of Na_2SO_4 may form at the NaCl surface. Subsequently, as suggested by the thermodynamic data (9) for the equilibrium between SO_4^{-2} and $S_2O_7^{-2}$ (i.e., $SO_4^{-2} + SO_3 \rightleftharpoons S_2O_7^{-2}$), the ratio of $Na_2S_2O_7$ to Na_2SO_4 at equilibrium should be of the order of 10:1 for a SO_3 concentration of 0.1 mole percent. Consequently, any Na_2SO_4 formed at the NaCl surface at 415°C should convert, essentially, to molten $Na_2S_2O_7$.

Formation of Solid Na_2SO_4

Between about 450° and 625°C, solid Na_2SO_4 (as shown by chemical and X-ray analyses) is the principal film product that forms on the NaCl surface when it is exposed to SO_2-O_2 gas mixtures containing about 0.1 mole percent SO_3. These films, unlike the $Na_2S_2O_7$ films at 420°C, are porous and are removed easily from the NaCl crystal upon cooling. This predominance of Na_2SO_4 over

$Na_2S_2O_7$ for this temperature range is in agreement with the Na_2SO_4-$Na_2S_2O_7$ concentrations as calculated from the thermodynamic data for the equilibrium between $S_2O_7^{-2}$ and SO_4^{-2} (9). For example, for a SO_3 concentration of 0.1 mole percent, the Na_2SO_4 concentration exceeds that of $Na_2S_2O_7$ at about 450°C and becomes increasingly larger with increasing temperature.

High pressure mass spectrometry showed that Cl_2 was the principal gaseous product produced during the reaction under anhydrous conditions while HCl is evolved when reasonable quantities of H_2O are present. Experiments showed that SO_3 was the key reactant rather than SO_2. For example, the rate of formation of Na_2SO_4 at 500°C was almost three orders of magnitude faster for the anhydrous system when the Pt catalyst was present. For both of these systems, the SO_2 levels were comparable but the SO_3 concentration was neglible for the system without the catalyst.

A typical curve illustrating the influence of flow rate upon the rate of weight gain is shown in Fig. 4. Both the anhydrous and the H_2O-containing systems gave similar types of curves except that the actual rates of weight gain were larger when H_2O was present. At the lower flow rates, the rates of weight gain were limited by the quantity of SO_3 available to maintain the reaction rate. For both systems, however, SO_2 was present in excess. At moderate and higher flow rates, the rates of weight gain became independent of flow rate.

The rates of weight gain were essentially constant with time for both systems. However, for comparable quantities of SO_3, the rates were almost an order of magnitude larger for the systems containing 0.1 mole percent H_2O than for the anhydrous systems. A typical curve for an anhydrous system is shown in Fig. 5 for the exposure at 465°C. Also, mass spectrometry results showed that for the respective reactions, the rates of formation of Cl_2 and HCl were essentially constant with time.

The rates of weight gain were first order with pressure of SO_3 for the anhydrous sytems and first order with H_2O content for those systems containing varying concentrations of H_2O. Typical curves are shown in Fig. 6. Likewise, the rates of formation of HCl were shown by mass spectrometry to be first order with H_2O concentration.

Arrhenius-type plots are shown for both systems in Fig. 7 where the data has been normalized to 0.1 mole percent SO_3. For the anhydrous systems, NaCl crystals were exposed to gaseous mixtures of SO_2-O_2 containing about 0.1 mole percent SO_3. The rate K, expressed in units of mg cm^{-2} sec^{-1}, is:

$$K = 0.6\ (P_{SO_3})\ \exp\ (-22,000/RT) \qquad [6]$$

where pressure is in units of atmospheres. The values for the activation energy and preexponential factor are quite small, being about 22 KJ Mol^{-1} K^{-1} and 0.6 mg cm^{-2} sec^{-1} atm^{-1}, respectively.
For the systems containing 0.09 mole percent H_2O, the rates of weight gain were almost an order of magnitude faster than for the anhydrous sytems, but the slope was essentially zero.

Even though the calculated activation energy for the anhydrous system is small and is similar to values usually observed for processes involving diffusion of gaseous reactants through a boundary layer, a boundary layer diffusion process does not seem likely because the measurements were made at flow rates where the rates of weight gain were independent of flow rate. Furthermore, the rates of weight gain of a NaCl single crystal increased rather sharply at about 630°C (to be discussed later), in contrast to that process for which boundary layer diffusion is limiting. For example, boundary layer diffusion rates should not change radically as the temperature was increased from 620° to 640°C. As a consequence, the large increase in rate near 630°C would not occur if boundary layer diffusion was the rate limiting process at 620°C. Therefore, it is proposed that the limiting process for the formation of a solid Na_2SO_4 film between 450° and 625°C, under anhydrous conditions, is the kinetics of the reaction of SO_3 at the NaCl surface.

Because the observed activation energy value was small, it is believed that no strong bonds are being broken. Such a low energy process has been proposed by Anderson for the formation of Na_2SO_4 (10). This process can be illustrated by the following equations:

$$SO_3 + (SO_4) \rightarrow (S_2O_7)_T \qquad [7]$$

$$(S_2O_7)_T + 2e \rightarrow (SO_4^{-2})_{TP} + SO_3 \qquad [8]$$

$$(SO_4^{-2})_{TP} \rightarrow SO_4^{-2}{}_{Tet} \qquad [9]$$

For example, SO_3 and a planar (SO_4) structure (produced from SO_2 and O_2) are absorbed upon the NaCl surface to form a S_2O_7 transition structure (i.e., $(S_2O_7)_T$). This transition species is not the pyrosulfate structure but instead involves connecting the SO_3 and (SO_4) species by a O-O bond. Rearrangement, Cl_2 oxidation (i.e., electron transfer), and loss of SO_3 leads to a trigonal pyramidal SO_4^{-2} ion (i.e., $(SO_4^{-2})_{TP}$). Then, Anderson suggested that the rate limiting step, which requires only a relatively small energy, may involve the transformation of this structure to the tetrahedral SO_4^{-2} ion (i.e., $SO_4^{-2}{}_{Tet}$).

Because $Na_2S_2O_7$ is observed at lower temperatures, loss of SO_3 prior to the rate limiting step may not be necessary. Therefore, an alternate, parallel process, which, presumably, can also occur with similarly low energy requirements, is also being considered (10). This alternate process can be illustrated by the following equations:

$$SO_3 + (SO_4) \rightarrow (S_2O_7)_T \qquad [10]$$

$$(S_2O_7)_T + 2e \rightarrow (S_2O_7^{-2})_{TP} \qquad [11]$$

$$(S_2O_7^{-2})_{TP} \rightarrow S_2O_7^{-2}{}_{Tet} \qquad [12]$$

For example, for this alternate process, the rate limiting step may be the transformation of the trigonal pyramidal $S_2O_7^{-2}$ ion to the tetrahedral $Na_2S_2O_7$ (reaction [12]). Any $Na_2S_2O_7$ so produced is in equilibrium with Na_2SO_4. Because Na_2SO_4 is the more stable species at these temperatures, the $Na_2S_2O_7$ will then decompose to give solid Na_2SO_4 at the NaCl surface.

As indicated previously (Fig. 7), the rates of weight gain with temperature for the the sytems containing 0.09 mole percent H_2O could be fitted to one straight line with a slope of essentially zero (at about 1.5×10^{-4} mg cm^{-2} sec^{-1}). Because the rate was constant with time, it is proposed that the controlling process involves transport of the diffusing species through a film of constant thickness. Presumably, the initial reaction to form an intermediate is faster than the subsequent decomposition of the intermediate to SO_4^{-2}. As a consequence, the initial reaction rate may decrease rapidly with time as the film thickness is increasing. Then, when the rate has decreased to that point where it is equal to the rate of decomposition of the intermediate (to form solid Na_2SO_4), the film thickness may remain constant. The overall process then would be controlled by transport through this film of constant thickness. No postulate can be put forward at this time to explain the observed lack of an activation energy for this process.

Formation of Molten Na_2SO_4-NaCl

Initial experiments showed that the rates of weight gain increased sharply (by at least an order of magnitude) when the single crystal NaCl temperature was raised above 625°C during exposure to SO_3-SO_2-O_2 atmospheres. Samples held at temperature above 640°C for short intervals and then quenched showed a molten film mixture of Na_2SO_4 and NaCl on the surface, as determined by sulfate and chloride analyses. This is in agreement with the phase diagram which shows that a eutectic is formed at about 625°C (11).

Similar results were obtained when solid mixtures of 1:1 Na_2SO_4 and NaCl were exposed to SO_3. For example, upon exposing this solid mixture to 0.1 mole percent SO_3 between 620° and 630°C, the mixture melted and the rate of weight gain above 625°C increased rather sharply by almost an order of magnitude. For data taken after relatively short times, the rate of weight gain remained essentially contstant at about 10^{-3} mg cm^{-2} sec^{-1} over the range of 650° to 850°C.

The rates of the reaction of SO_3 with the NaCl in the molten 1:1 mixture of NaCl and Na_2SO_4 were determined by exposing the mixture to 0.1 mole percent SO_3 between 700° and 750°C. The data, normalized to 0.1 mole percent SO_3, is shown in Fig. 8. The linear curve indicates parabolic behavior with a rate constant of 7.0×10^{-4} mg^2 cm^{-4} sec^{-1}. From this, a diffusion coefficient of about 7×10^{-5} cm^2 sec^{-1} is calculated.

It is proposed, therefore, that for temperatures above 625°C, SO_3 reacts with NaCl at its surface to produce Na_2SO_4 which can then form a molten phase with the NaCl. The process becomes diffusion controlled as SO_3 reacts with NaCl in the melt, producing additional Na_2SO_4.

Sodium Chloride Particle Conversion Time

Assuming a rate of about 10^{-3} mg cm^{-2} sec^{-1} for the reaction of NaCl with SO_3 at 800°C, an order of magnitude estimate can be made for the time required to consume NaCl particles (forming Na_2SO_4)

under simulated gas turbine operating conditions. For example, a 100 µm NaCl cube particle (i.e., 100 µm x 100 µm x 100 µm) upon reaching 800°C when exposed to a combustion atmosphere containing about 0.01 mole percent SO_3, would require more than 2 hr to be converted to Na_2SO_4. This rather lengthy time interval suggests that the film formed on the component parts should consist primarily of NaCl with only traces of Na_2SO_4. This seems to be contrary to what is actually observed. As a consequence, therefore, proposed processes involving impaction of NaCl particles upon the component parts with subsequent reaction to form Na_2SO_4 are assumed to be of less significance than processes involving a conversion to Na_2SO_4 in the hot combustion stream with subsequent deposition (12).

SUMMARY

1. A series of product films were obtained upon exposure of NaCl to SO_3 in SO_2-O_2 gas mixtures: (1) solid $NaCl \cdot SO_3$; (2) molten $Na_2S_2O_7$; (3) solid Na_2SO_4; and (4) a molten mixture of Na_2SO_4 and NaCl.

2. Experimentally measured rates indicated that processes 1, 2, and 4 of the above were mass transport controlled. The formation of solid Na_2SO_4 (process 3), however, was a reaction kinetic process with a small activation energy of about 22 KJ Mol^{-1} K^{-1}. Mechanisms for this process, involving the transformation of a S_2O_7 transition structure to a tetrahedral SO_4^{-2} ion, are proposed.

3. The relatively slow rates at 800°C suggest that process 2 where Na_2SO_4 forms in the hot combustion atmosphere and then deposits on the turbine component parts may be of greater significance than process 1 where NaCl impacts on the component surfaces and reacts there to form Na_2SO_4.

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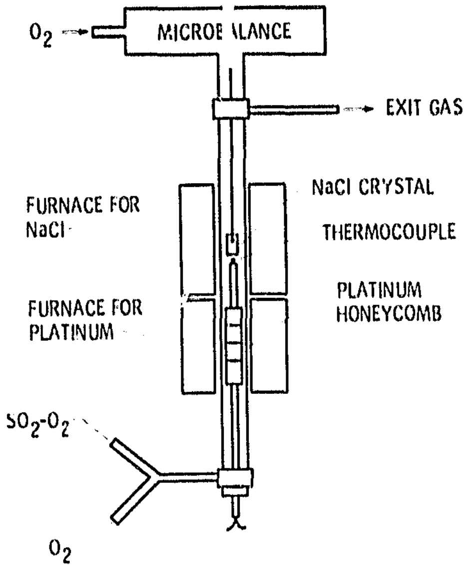

Figure 1. - Schematic of the thermogravimetric apparatus.

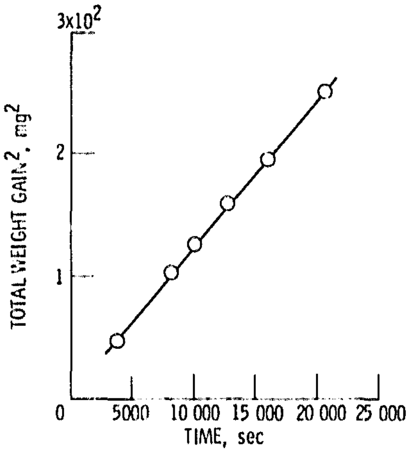

Figure 2. - Plot of (weight gain)^2 with time for the exposure of single crystal NaCl to 0.1 mole percent anhydrous SO_3 at 150° C.

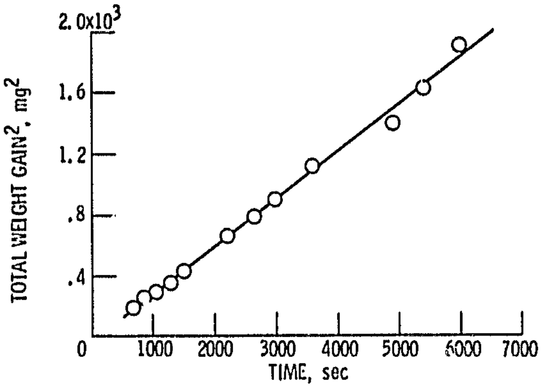

Figure 3. - Plot of (weight gain)^2 with time for the exposure of single crystal NaCl to 0.1 mole anhydrous SO_3 at 415° C.

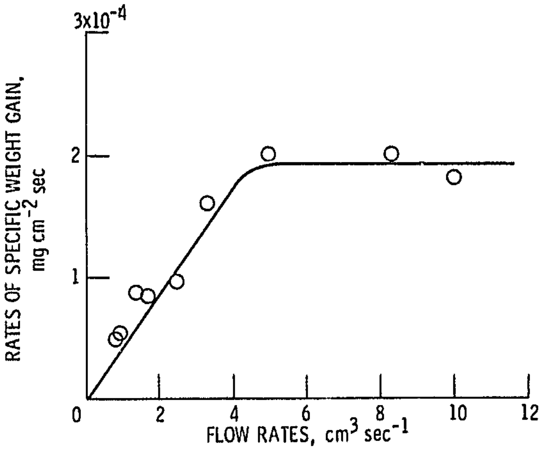

Figure 4. - Plot of rates of specific weight gain with flow rates for the exposure of single crystal NaCl to 0.3 mole percent H_2O and 0.01 mole percent SO_3 at 550° C.

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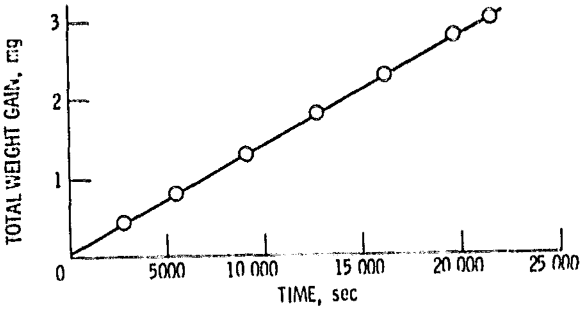

Figure 5. - Plot of total weight gain with time for the exposure of single crystal NaCl to 0.1 mole percent anhydrous SO_3 at 465° C.

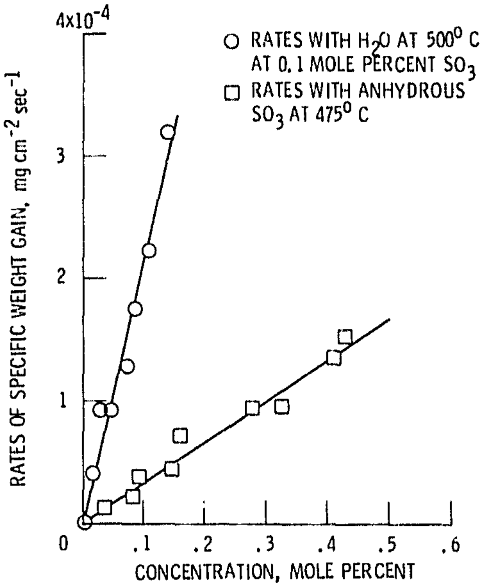

Figure 6. - Plot of rates of specific weight gain with concentration for the exposure of single crystal NaCl to SO_3 and H_2O.

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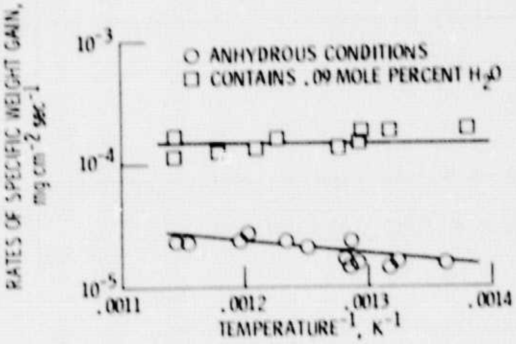

Figure 7. - Plot of rates of the specific weight gain with temperature^{-1} for the exposure of single crystal NaCl to 0.1 mole percent SO_3 under anhydrous and moisture conditions.

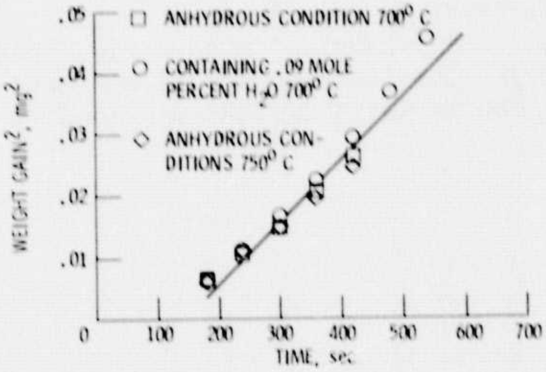

Figure 8. - Plot of (weight gain)^2 with time for the exposure of a molten mixture of 1:1 Na_2SO_4 and NaCl to 0.1 mole percent SO_3.