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COMPARISON OF OUTGASSING RATES FROM STAINLESS STEEL, ALUMINUM, AND COPPER AFTER BAKEOUT UNDER HIGH VACUUM AND ATMOSPHERIC PRESSURE

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ABSTRACT

Outgassing rates of vacuum vessels consisting of stainless steel, copper, and aluminum (inside surface area 7500 cm^2) at $25^{\circ}C$ were measured after bakeout at 50 to $150^{\circ}C$ (up to $250^{\circ}C$ for s.s.). For stainless steel the outgassing rates obtained after vacuum bakeout (about 1×10^{-12} Torr liters/sec cm^2) agree well with those found by other investigators. Similar values were found for copper. Much lower values of the outgassing rates were obtained for aluminum (0.3 to 1.8×10^{-13} Torr liters/sec cm^2). The outgassing rates obtained after a 2 h bakeout in air at atmospheric pressure followed by a 18 h vacuum bakeout are significantly lower than those after a vacuum bakeout; in the case of stainless steel an air bakeout gives a permanent reduction of the outgassing rate.

INTRODUCTION

It is well known that vacuum systems have to be baked at elevated temperatures if clean surfaces and low outgassing rates are required. A summary of the processes occuring during bakeout is given for instance by Redhead et al[1].

Most experimental work was done for stainless steel. For this material the outgassing rates obtained after bakeout are between 10^{-12} and 10^{-13} Torr liters/sec cm^2. Calder and Lewin[2] showed that after bakeout at 360°C for one day the outgassing rate of stainless steel is mainly given by the diffusion of H_2 out of the bulk. 1968 it could be shown independently by Strausser[3] and by a group in our laboratory[4,5] that much lower bakeout temperatures (150°C, and 80 to 250°C respectively, applied for one day) are sufficient to remove the adsorbed water vapour and to give low outgassing rates comparable with those obtained after bakeout at higher temperatures.

Another technique - bakeout in air at atmospheric pressure followed by a vacuum bakeout - was investigated by Young[6] and by Samuel[7]. This method which, apparently, gives lower outgassing rates than those obtained after vacuum bakeout is used, for example for evacuation of the CERN storage rings.

Less work was done for aluminum and copper. After a 15 h vacuum bakeout at 250°C Young[6] found outgassing rates of 4×10^{-13} Torr liters/sec cm^2, for aluminum. Similar results were obtained in our laboratory[5] for a 24 h bakeout at 80 to 250°C.

For copper samples, baked under vacuum at 175 to 400°C for several hours, Zhilnin et al[8] gave extrapolated outgassing rates between 10^{-11} and 10^{-14} Torr liters/sec cm^2.

The aim of this work is to compare outgassing rates of stainless steel, copper, and aluminum after bakeout. Bakeout was done in vacuum as well as in air under atmospheric pressure followed by a vacuum bakeout. The bakeout temperatures investigated were between 50 and 150°C (for stainless steel up to 250°C), the bakeout time was choosen between 5 and 20 h.

EXPERIMENTAL TECHNIQUES

Fig. 1 shows the apparatus used for the measurement of outgassing rates. The measuring device between sample and pump consists of two volumes V_1 and V_2 separated by an orifice of known conductance which can be set to 0.09 and 1.5 liters/sec. The pressures p_1 and p_2 in V_1 and V_2 were measured by means of BA-gauges. Thoriated Iridium filaments operated with 40 μA emission were used, in order to minimize the influence of the gauges. In a separate work[9] it could be shown that under these conditions the pumping speed of the gauges for H_2 is less than 0.1 liters/sec. The gas composition of the outgassing rate was determined by means of a Quadrupole residual gas analyser. The inside surface area of the samples was 7500 cm^2 (geometrical value). The bakeout temperature of the apparatus could be choosen between 50 and 250°C. For evacuation a sputter ion pump with 80 liters/sec was used; rough pumping was performed by means of cryosorption pumps.

The gas flow from the sample into the measuring device could be interrupted by a valve. Opening and closing this valve we get pressure differences Δp_1 and Δp_2. The outgassing rate is obtained from Eq. (1):

$$Q = L\ (\Delta p_2 - \Delta p_1) \approx L\Delta p_2.$$

The gauges were calibrated by the dynamic expansion method and the accuracy of N_2-equivalent pressure readings was deemed to be better than 20 percent.

The samples were manufactured from 2 mm thick stainless steel sheets (Deutsche Werkstoff Nr. 4301; $C \leq 0.06$, Cr 18, Ni 9), 4 mm thick aluminum sheets (Al 99,5; $Si \leq 0.3$, $Fe \leq 0.4$, $Ti \leq 0.03$, $Cu \leq 0.05$) and 2 mm thick copper sheets (E-Cu). The samples were rinsed with acetone and baked under vacuum for 20 h at 200°C. At the beginning of each individual experiment the sample was filled with 760 Torr moist air (> 96 percent relativ humidity, 23°C) for 30 minutes. In the case of vacuum bakeout the sample was now evacuated; bakeout was started after a pressure of 10^{-3} Torr was reached. In the case of atmospheric bakeout the sample was baked in air for 2 h at 100°C, then evacuation was started and bakeout was continued for another 3 or 18 h. The measurement of the outgassing rate was taken one day after switching off the ovens, i.e. more than 10 h after the apparatus had reached room temperature again. After the measurement the sample was again exposed to moist air and the next experiment was started. After a series of typically 5 measurements the sample was exposed to dry nitrogen and removed from the system, and another sample was tested. After approximately one month the first sample was mounted again and the measurement was continued.

RESULTS

After bakeout and cooling down to room temperature the pressure in V_2 was between 5×10^{-9} and 5×10^{-8} Torr. The pressure difference Δp_2 obtained by opening and closing the valve between sample and measuring device was between 1.4×10^{-10} and 1×10^{-8} Torr (depending on the sample and the bakeout conditions). As the surface of the measuring device was much smaller than that of the sample, most of the outgassing rate of the measuring device was due to outgassing of the BA-gauge and the RGA (a similar observation was reported by Strausser[3]). The outgassing rates above 5×10^{-13} Torr liters /sec cm^2 could be measured with a relative accuracy of better than 15 percent; outgassing rates below 5×10^{-14} Torr liters/sec cm^2 have about 30 percent relative accuracy.

Tab. I shows the outgassing rates of stainless steel, copper, and aluminum obtained after a 20 h vacuum bakeout at 50, 100, and 150°C (250°C for s.s.). Data for the 100°C bakeout are average values from at least three experiments.

Tab. II gives the residual gas composition of the outgassing rates obtained after the 100°C vacuum bakeout. The absolute accuracy of residual gas composition for various runs was approx. 5 percent.

Tab. III gives the outgassing rate of the various gases observed (corrected for the individual conductance) for the 100°C vacuum bakeout.

Tab. IV gives N_2-equivalent outgassing rates obtained after 2 h bakeout in air at atmospheric pressure followed by a 3 or 18 h vacuum bakeout. The bakeout temperature was again 100°C. For Cu and Al the residual gas composition of the outgassing rates was similar to that given in Tab. II except of more CO_2 (30 to 40 percent) and less CO. In the case of stainless steel only about 23 and 63 percent H_2 could be detected for the 5 h and 20 h bakeout respectively; the rest was CO and CO_2.

In the case of stainless steel a vacuum bakeout at 100°C (20 h) was performed after the data of Tab. IV were measured. The N_2-equivalent outgassing rate was found to be 1×10^{-13} Torr liters/sec cm^2 (i.e. the same value as obtained after air bakeout). This means that an air bakeout at atmospheric pressure reduces the outgassing rate permanently.

CONCLUSION

(1) For stainless steel the measured values of the outgassing rate and their residual gas composition (mostly H_2) obtained after vacuum bakeout agree well with those found in literature[2-6] (partly measured after bakeout at noticeably higher temperatures).

(2) The outgassing rates found for Cu are approximately the same as for stainless steel; the outgassing rates of aluminum are more then one order of magnitude lower than those of stainless steel and copper.

(3) Bakeout at $50^{o}C$ for 20 h reduces the H_2O-content of the outgassing rate to less than 5 percent; however, after such a bakeout the outgassing rates for the other gases are noticeably higher than after a bakeout at a temperature $> 100^{o}C$.

(4) After a vacuum bakeout, for stainless steel the outgassing rate is mainly due to H_2 ($>$ 80 percent), in the case of copper to CO ($>$ 70 percent) and in case of Al to $>$ 50 percent CO.

(5) Applying the same bakeout time we get much lower values of the outgassing rate after air bakeout than after vacuum bakeout.

(6) In the case of stainless steel and copper the 2 h bakeout in air at atmospheric pressure followed by the 3 h vacuum bakeout gives much lower values of the outgassing rate than the 20 h vacuum bakeout.

(7) For stainless steel air bakeout reduces the outgassing rate permanently: After air bakeout the higher values of the outgassing rates after a vacuum bakeout could not be achieved again. Subsequent exposure to moist air followed by vacuum bakeout gives again 1×10^{-13} Torr liters /sec cm^2, i.e. much lower values compared with the original data of Tab. I (1×10^{-12} Torr liters/sec cm^2).

TABLE I. N_2-equivalent outgassing rates of stainless steel, copper, and aluminum after a 20 h vacuum bakeout at 50, 100, and 150°C (250°C for stainless steel).

Outgassing rates in units of 10^{-13} Torr liters/sec cm^2 (N_2-equivalent)				
	50°C	100°C	150°C	250°C
Stainless steel	13	10	10	7
Copper	26	11	6	-
Aluminum	1.8	0.4	0.3	-

TABLE II. N_2-equivalent outgassing rates and residual gas compositions (corrected for RGA-sensitivities) of stainless steel, copper, and aluminum after a 20 h vacuum bakeout at 100°C.

	N_2-equivalent outgassing rate (Torr liters/sec cm^2)	Residual gas composition (percent)				
		H_2	CH_4	H_2O	CO	CO_2
Stainless steel	10×10^{-13}	82	0.5	1	13	2
Copper	11×10^{-13}	4	1.5	0	76	17
Aluminum	0.4×10^{-13}	17	3	0	52	25

TABLE III. Outgassing rates of stainless steel, copper, and aluminum after a 20 h vacuum bakeout at 100°C (corrected for individual conductances).

Outgassing rates in units of 10^{-13} Torr liters/sec cm^2.					
	H_2	CH_4	H_2O	CO	CO_2
Stainless steel	74	0.16	0.30	3.2	0.38
Copper	1.73	0.23	-	8.9	1.6
Aluminum	0.30	0.02	-	0.24	0.10

TABLE IV. N_2-equivalent outgassing rates of stainless steel, copper, and aluminum after a 2 h bakeout in air at atmospheric pressure (100°C) followed by a 3 or 18 h vacuum bakeout.

N_2-equivalent outgassing rates in units of 10^{-13} Torr liters/sec cm^2

	5 h	20 h
Stainless steel	4.2	1.0
Copper	5.8	3.0
Aluminum	0.8	0.28

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FIGURE CAPTIONS

Fig.1. Apparatus used for the measurement of the outgassing rates.

V Valve between measuring device and sample

V_1 Volume V_1 with pressure p_1

V_2 Volume V_2 with pressure p_2

L Conductance of the orifice

S Pumping speed of the sputter ion pump

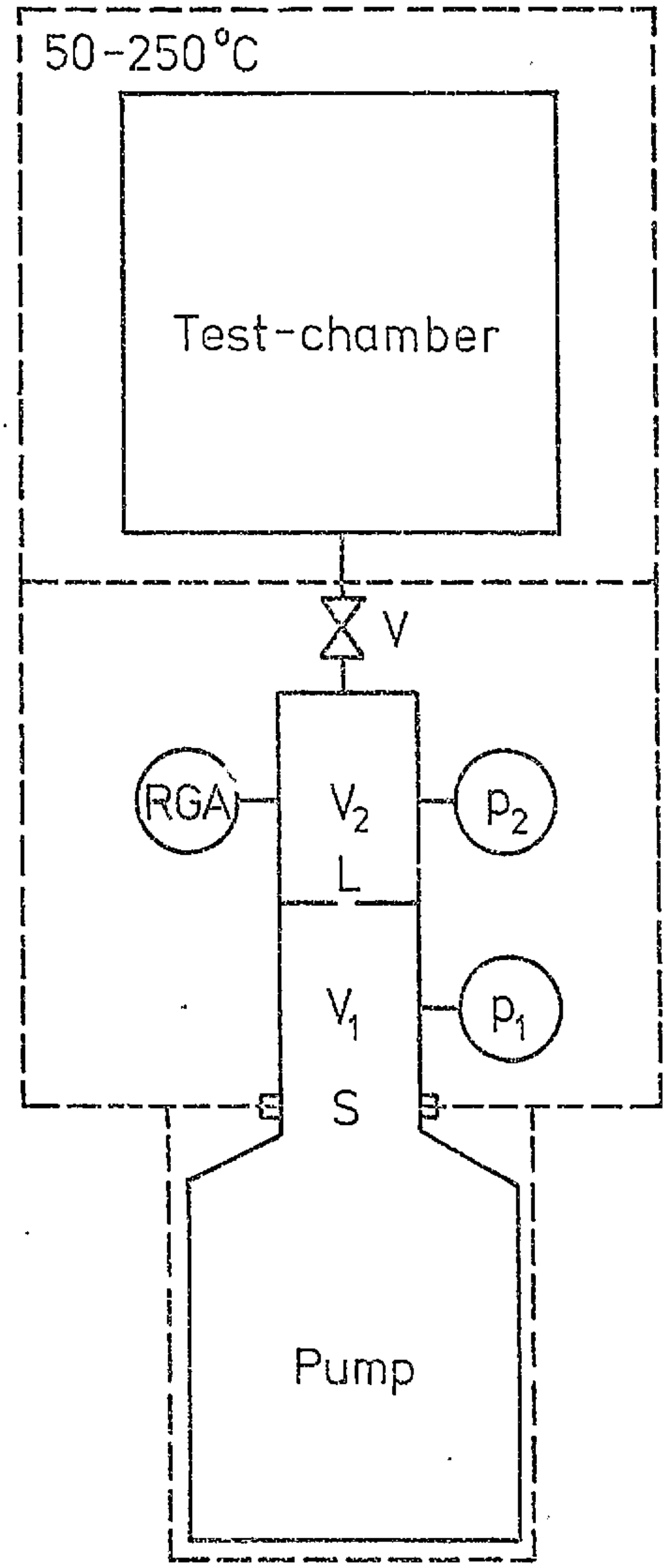

Fig.1

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