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AN AUTOMATIC OPERATION OF VACUUM SYSTEM FOR ELECTRON MICROSCOPIC PREPARATIONS

by

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(with 1 Table and 2 Text-figures)

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The electron microscopic observation of geologic or paleontologic samples usually applies single stage or two stage replica technique (HONJO and FISCHER, 1965, HONJO, 1968). In the replica preparation procedure, the vacuum evaporation of metals for shadowing technique, or vacuum deposition of carbon film is found to be a neck throughout the entire flow of the preparation procedures. The most of the time which is consumed during the vacuum evaporating preparation is indeed devoted to obtain high vacuum at pressures about 10^{-5} torr. or less. The scanning electron microscopy also requirs coating of samples with vacuum evaporation technique and usually a great amount of the processing is required for such research works (HONJO and OKADA, 1968).

Therefore, the efficiency of the entire preparation procedure is said to be decided by the efficiency of the vacuum plant for evaporation. This situation is analogous to the productivity of an automobile factory which depends on its steel-press capacity.

The final quality of replica or metalic coating much depends on the vacuum technique. Particularly, an operator should endeavor to prepare a better vacuum as possible in a shadowing procedure to secure the finer grainity of metals or carbon which plays a decisive role in the demarkation of electron photomicrographs. Or, if the pressures are not low enough, the mechanical strength of a carbon film will be inferior due to the trapped gas molecules among the deposited carbon particles. An instrument to provide a higher vacuum within a shorter time has been thus required by electron microscopic laboratories.

Recent development of high vacuum technology has provided us many methods to obtain such high vacuum. Among them, one of the most classical systems, i.e., a combination of a rotary pump and an oil diffusion pump, is found to be the most

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handy method for an electron microscopic as well as a scanning electron microscopic preparation in terms of the economy and the simplicity of the operation. The evaporation of metals or carbon is satisfactorily conducted under the pressures of 10^{-5} torr. or lower. This is approximately the lowest paressurs which can be attained by the combination of rotary and oil diffusion pumps without high temperature baking technique.

Basically, the speed of evacuation depends on the size of a space to be evacuated, the pumping speeds, the leak and out-gas factors and other miscellaneous factors such as drags caused by the complex apparatus which are installed in a belljar. The evacuation speed of above mentioned system is usually reasonable. The use of an over-sized oil diffusion pump significantly improves the evacuation speed. However, the installation of an efficient liquid nitrogen cooling trap is essential to trap the backstream of oil vapor from hot diffusion oil.

Besides such basic factors, the timing to switch the system from the *rough evacuation* to the *high vacuum* evacuation is an important factor for the total efficiency. Or, a device to inform an operator that the pressures are low enough to operater vacuum evaporation works, helps to minimize the waste of time.

Misoperation of a high vacuum instrument is easily comitted, mainly because the valve operation is somewhat complex for a novice operator. A typical one is to open the *by-pass valve* during the *fore valve* is left open and diffusion oil remains hot, or to open the *main valve* when the bell-jar pressures are high. Such accident leads to contaminate samples or to spoil diffusion oil.

Based on the above consideration, we designed an automatic vacuum unit herewith described. The vacuum unit is operated with the six manual push-button type switches. They are STANDBY, START, OPEN, RESET, STOP and CHAMBER LEAK switches. The all valves are properly opened and closed by electronically controlled pneumatic drivers. The *main valve* is opened or closed at the proper pressures where the oil diffusion pump works with the maximam efficiency. When the bell-jar space is evacuated down to the desired pressures, preset relay system rings chime sounds to call attention.

The fail-safe circuit detects wrong setting of valves and informs it to an operator. Especially, the fail-safe system protects the unit from admitting air into the hot diffusion pump throughout the entire procedure. Therefore, the instrument can be operated safely with a constant efficiency by an operator who entirely lacks the backgroun of vacuum technique. The unit can be switched to manual operation immediately if desired at any phase of the automatic operation.

The sequence chart of the automatic operation of our vacuum unit is shown in Table 1. The explanation of the detail motion of the unit during outomatic operation is as fallows.

0 The entire electricity surply for the instrument is stopped (except to the air compresser). All valves are closed except to the *pump leak valve* is left

Table 1. Sequence diagram of the automatic vacuum unit.

No.	Switch	Signal	Water	Air	RP	DP	P-L	C-L	BV	FV	MV	PM	PHG	Chime	
0	STANDBY		×	●	×	×	●	×	×	×	×	×	×	×	STOP ELECTL
1		PSW-DR_1ON	●	●	●	×	●	×	×	×	×	×	×	×	
2	CHAM. LEAK	DR_1 SR_1, S_2	●	●	●	×	×	×	×	×	×	●	×	×	CHAM. LEAK. PL
3			●	●	●	×	×	●	×	×	×	●	×	×	
4	START		●	●	●	×	×	×	●	×	×	●	×	×	ROUGH EVAC.
5		PM-TY_1ON	●	●	●	●	×	×	●	●	×	●	×	×	DP. ON
6		TY_1-DR_3ON	●	●	●	●	×	×	×	●	×	●	×	×	
7		DR_3-MV	●	●	●	●	×	×	×	●	●	●	●	×	HIGH VACUUM EVACUATION
8	EVAP	PHG	●	●	●	●	×	×	×	●	●	●	●	●	
9	OPEN		●	●	●	●	×	×	×	●	●	●	●	×	EVAPORATION
10	CHAM. LEAK	S_1, S_2	●	●	●	●	×	×	×	●	×	●	×	×	CHAM. LEAK. PL
11	RESET	-DR_4ON	●	●	●	●	×	●	×	×	×	●	×	×	
12		DR_4	●	●	●	●	×	●	×	×	×	●	×	×	
13		PM-DR_3ON	●	●	●	●	×	×	●	×	×	●	×	×	ROUGHING
14		DR_3	●	●	●	●	×	×	×	●	×	●	×	×	
15		PHG	●	●	●	●	×	×	×	●	●	●	●	×	HIGH VACUUM EVACUATION
16	EVAP		●	●	●	●	×	×	×	●	●	●	●	●	
17	OPEN		●	●	●	●	×	×	×	●	●	●	●	×	EVAPORATION
18	CHAM. LEAK	S_1, S_2	●	●	●	●	×	×	×	●	×	●	×	×	CHAM. LEAK. PL
19	RESET	-DR_4ON	●	●	●	●	×	●	×	●	×	●	×	×	
20		DR_4	●	●	●	●	×	●	×	×	×	●	×	×	
21		PM	●	●	●	●	×	×	●	×	×	●	×	×	
22	STOP	-TY_2ON	●	●	●	●	×	×	×	●	×	●	×	×	
23		TY_2-DR_5ON	●	●	●	×	×	×	×	●	×	×	×	×	D.P. off
24		DR_5	●	●	●	×	×	×	×	×	×	×	×	×	
0			×	●	×	×	●	×	×	×	×	×	×	×	STOP E. SOURCE

● : being opened or operated. × : being closed or out of operation. RP : rotary pump ; DP : oil diffusion pump ; BV : by-pass valve ; FV : fore valve ; MV : main valve ; PM : Pilani meter ; PHG : Philips gauge ; PSW : pressure switch ; DR_n : delay relays ; TY_n : timers ; PL : pilot lamp.

open.

0.1 The STANDBY switch is manually thrown-in, then

0.1.1 the rotary vacuum pump starts, and

0.1.2 the *pump leak valve* is closed gradually. The pneumatic delay reserver tank NDR, shown in Fig. 2, slows down the dirver. When the air pressures are not high enough, the sequence stays here.

0.1.3 Cooling water is admitted into the water jacket of oil diffusion pump by opening a magnetic valve through a *water pressure* switch (PSW in table 1), then

0.1.3.1 delay relay no. 1 (DR_1 in table 1) holds the action of 15 seconds.

1.2 When 0.1.3.1 is completed,

1.2.1 the electric surply to the Pilani meter is turned on, and

1.2.2 the STANDBY pilot lamp is turned on, and

1.2.3 a pilot lamp indicates that the air is ready to be admitted into the bell-jar, in case the main valve is closed (c s w in Fig. 2).

Out of sequence: The CHAMBER LEAK switch is thrown-in to let the air into the bell-jar through the pneumatic *chamber leak valve*. The leak rate is variable on the demand. Lift the bell-jar. Preparation for evaporation works in the bell-jar such as to set samples, carbon arcs, etc. The bell-jar is then closed.

3.4 Throw-in the START switch, then

3.4.1 the *chamber leak valve* is closed, and

3.4.2 the *by-pass valve* is opened.

4.5 When the Pilani meter (PM) indicates the pressures of 6×10^{-2} torr. (set position is variable upon the back presures of the oil diffusion pump),

4.5.1 the *fore valve* is opened, and

4.5.2 the electric heater for the oil diffusion pump is turned on, and

4.5.3 the timer no. 1 (TY_1) holds the next action for the intervals between 10 to 40 minutes (enough to heat up the diffusion pump).

5.6 The timer no. 1 actuates to close the *by-pass* valve after 4.5.3 is completed,

5.6.1 the delay relay no. 3 (DR_3) holds the action for 10 seconds.

6.7 After DR_4 completes the interval,

6.7.1 the *main valve* is opened, and

6.7.2 the electric source for Philips gauge is turn on.

7.8 The Philips gauge stays on action.

8 At the preset vacuum on the Philips gauge such as 2×10^{-5} torr. (set position is variable), the chime rings for attention. (Turn the snap-switch off manually to stop the chime.)

8.1 The evaporation heater source (200 A, 30 V at the maximum) is turned on through a magnetic relay.

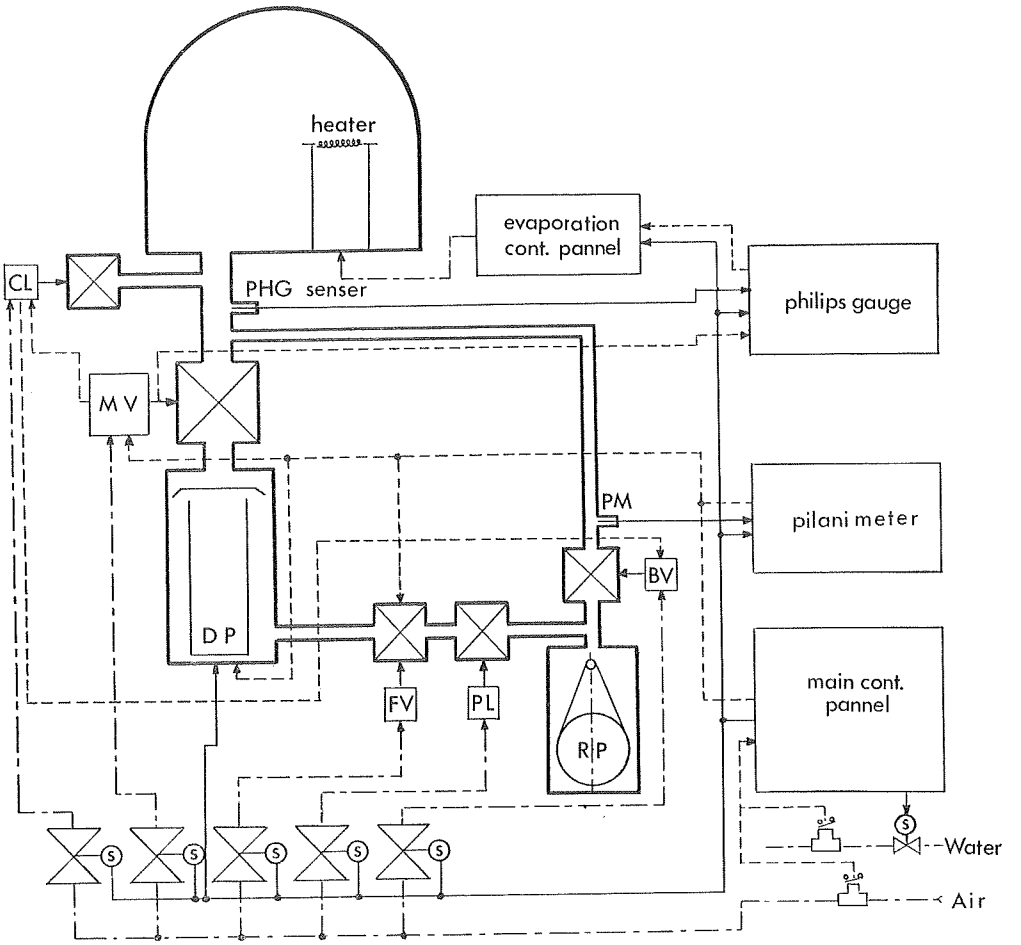

Fig. 1
Block diagram of automatic vaccuum unit. Solid lines; control. Broken lines; checking. Dot-dash-lines; operation.

Out of Sequence: Vacuum evaporation works.

9.10 To open the bell-jar, throw-in the OPEN switch, then

9.10.1 the Philips gauge is shut off, and

9.10.2 the evaporation heater source is shut off, and

9.10.3 the *main valve* is closed.

9.10.3.1 The fail-safe switch (fsw in Fig. 2) of the *main valve* is actuated by closing of the *main valve*, and a pilot lamp indicates that the *chamber leak valve* is ready to be opned, and

9.10.3.2 the *chamber leak valve* is ready to be opened.

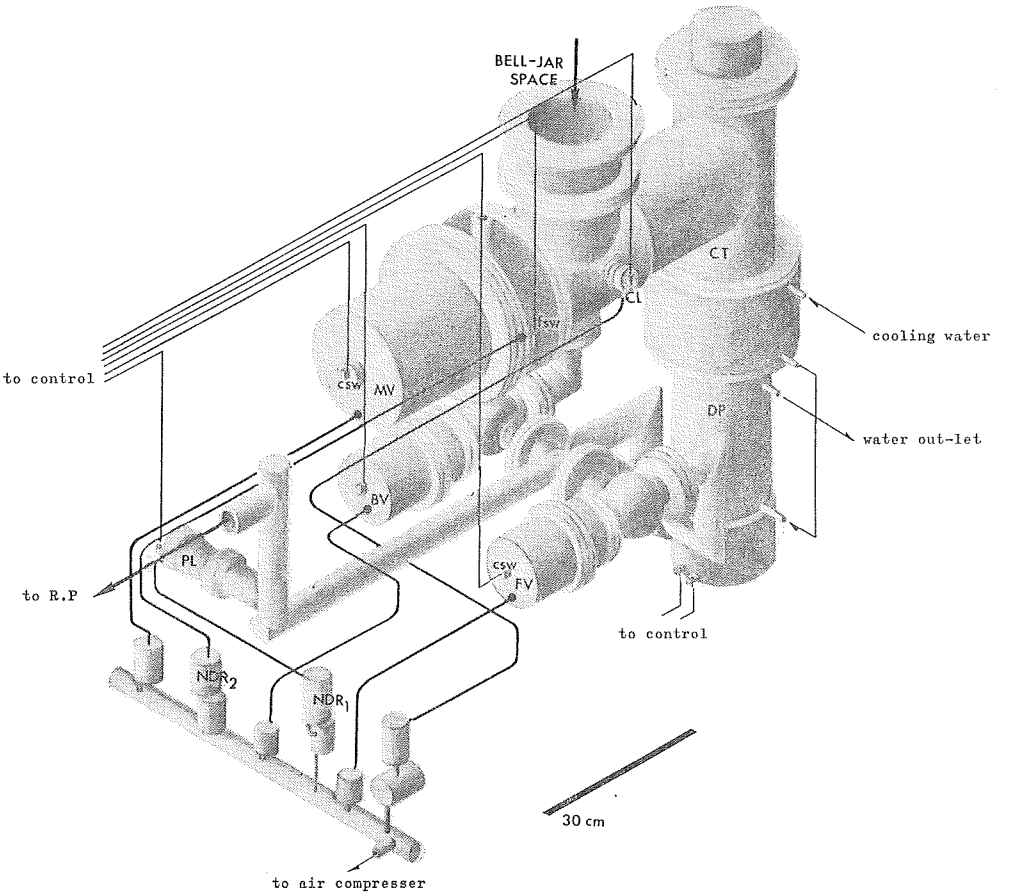

Fig. 2
Arrangement of the valves and manifolds of the automatic vaccum unit. Bold lines; pneumatic operation. Thin lines; electric control. csw; check switch, fsw; fail safe switch, CT; liquid nitrogen cold trap, NDR_n; pneumatic delay relays.

Out of sequence: Throw-in the CHAMBER LEAK switch to open the bell-jar. Preparation for evaporation works. Then close the bell-jar.

11.12 Throw-in the RESET switch, then

11.12.1 the *chamber leak valve* is closed, and

11.12.2 the *fore valve* is closed,

12.13 The delay relay no. 4 (DR_4) holds action for 5 seconds, and

12.13.1 the *by-pass valve* is opened when DR_4 completes the interval.

13.14 Return to 12, and repeat the sequence from 12 through 22.

22.23 To stop the vacuum unit, throw-in STOP switch, at any time when the *fore*

valve is left open.

22.23.1 the *main valve* and the *by-pass* valve are closed if they are, or one of them is, left open.

22.23.2 the heater source of the oil diffusion pump is shut off, and

22.23.3 the Philips gauge is shut off, and

22.23.4 the Pilani meter is shut off, and

22.23.5 the timer no. 2 (TY_2) holds action for any preset interval between 20 to 40 minutes.

23.24 The timer no. 2 actuates to close the *fore valve* after TY_2 completes the interval, then

23.24.1 the delay relay no. 4 (DR_4) holds action for 5 second.

23.24.1.1 After DR_4 completes the interval, the rotary pump is stopped, and

23.24.1.2 the *pump leak valve* is opened, and

23.24.1.3 water supply for the oil diffusion pump is shut off by closing the magnetic valve, and the sequence returns to 0.

Our automatic vacuum evaporator is equipted with a 4 inch diameter, 400 1/sec., oil diffusion pump and a rotary vacuum pump of 300 1/min. capacity. The bell-jar space is approximately 56 l. It takes approximately five minutes for rough evacuation to 6×10^{-2} torr. and less than ten minutes to evacuate the bell-jar space down to the pressures of 2×10^{-5} torr. A shadowing and carbon film deposition on plastic first replicas or rotary metalic coating on the samples for scanning electron microscopic study, can be operated approximately every thirty minutes at the optimum use.

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