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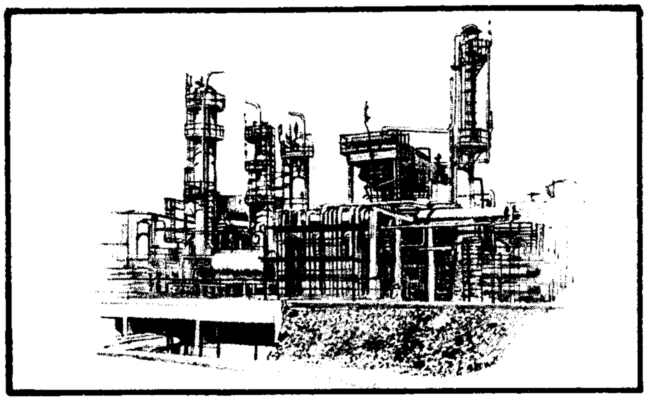

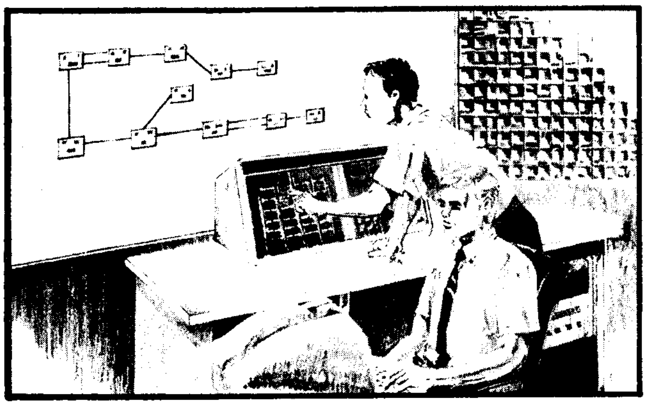

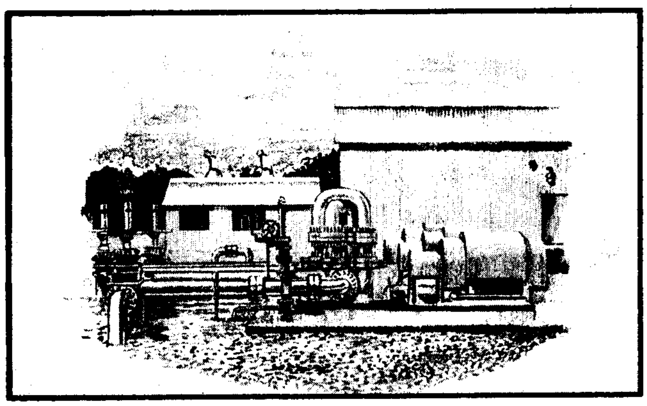

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FINAL REPORT
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by

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OPTIMUM OUTGASSING CYCLES FOR ALUMINUM AND STAINLESS STEEL

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ABSTRACT

Outgassing rates were measured for a modified 7075T6 aluminum and 304L stainless steel, for two degrees of finish, at temperatures of 285°F to 392°F, for times of 0.5 to 72 hours. The results were analyzed to determine optimum time and temperature cycles for outgassing. Optimum cycles were determined with and without the limitations of a graph. The graph related allowable time at temperature to a 5% or less reduction in room temperature properties of 7075T6 aluminum.

For aluminum, within the limits set by reduction in mechanical properties, the optimum cycle tested was 40 hours at 285°F. Disregarding the limits, optimum outgassing was achieved at the highest temperatures and longest times tested, for both aluminum and stainless steel. Aluminum outgassed less than half as much as stainless steel. The smoother finishes gave 20% - 35% lower outgassing rates initially, but this advantage decreased to 0% - 10% at 72 hours.

I. INTRODUCTION

During May, 1970, NASA-MSC directed Houston Research to perform Task Order No. 19 under Contract NAS9-6698. The objective was to determine the optimum time and temperature cycle for outgassing of a modified 7075T6 aluminum, and 304L stainless steel conforming to ASTM A276. A major constraint was that the chosen cycle had to lower the room temperature mechanical properties of the aluminum no more than 5%. This constraint was furnished by NASA-MSC as a graph of allowable time at temperature vs. temperature.

The optimum outgassing cycle was defined as the one which would remove adsorbed and absorbed contaminants to the greatest extent. The optimum cycle would achieve the goal of the least possible contamination of lunar samples while they are being transported in the Apollo lunar sample return container.

Specifically, this task required measurement and analysis of outgassing rates for 32 RMS and 63 RMS finishes of the aluminum, and 16 RMS and 63 RMS finishes of the stainless steel. The outgassing data were required at pressures less than 1×10^{-4} Torr, at temperatures of $285^{\circ}F$ to $392^{\circ}F$, for times up to 72 hours.

II. SAMPLE PREPARATION AND TEST APPARATUS

SAMPLE PREPARATION

Bulk samples of the aluminum and stainless steel were furnished by NASA-MSC. The bulk samples were then cut into a number of conveniently sized test samples. The test samples were right rectangular prisms approximately 0.25 cm by 1 cm by 8 cm (0.1 X 0.5 X 3 inches). They were machined and polished to the desired finishes. The aluminum samples were finished to 32 RMS and 63 RMS. The stainless steel samples were finished to 16 RMS and 63 RMS.

The degassing of vacuum system materials is accomplished in two steps: (a) preparation, which includes all manipulations performed with the material prior to its mounting in the vacuum chamber; and (b) processing of the mounted parts in the system in order to achieve and maintain a low pressure.

The aims of the preparation step are to free the material from all gases or gas-evolving substances, and to keep them in this clean condition until the part is mounted and the pumping of the system starts. It is well known from technical experience as well as from scientific experiments that the gas content (both adsorbed and absorbed) of the materials may vary widely according to their previous treatment.

Therefore, a cleaning procedure based on the Apollo lunar sample return container cleaning procedure (ALSRC-1574-2) was used. High purity solvents were employed throughout. The cleaning steps were as follows:

(1) Scrub with 1% Alconox, using a fiber bristle brush.

(2) Scrub with Freon (1,1,1 trichloro - 2,2,2 trifluoroethane) using a Teflon brush.

(3) Rinse with Freon and drain thoroughly.

(4) Scrub with isopropyl alcohol, using a Teflon brush.

(5) Rinse with distilled water and drain thoroughly.

(6) Rinse with a one-to-three mixture of isopropyl alcohol and benzene and drain thoroughly.

(7) Rinse with Freon and drain thoroughly.

(8) Rinse with a one-to-three mixture of isopropyl alcohol and benzene and drain thoroughly.

(9) Dry for 30 minutes at room temperature, protected from dust, supported at three contact points by a clean watch glass.

(10) Same as step (9) except support on reverse side.

At the end of the drying period, the sample was inserted into the outgassing apparatus using a pair of tweezers which had been similarly cleaned.

APPARATUS

Figure 1 is a schematic diagram of the apparatus used. Outgassing rates were determined by measuring the rate of pressure rise (dP/dt) in a closed evacuated chamber containing the sample. The perfect gas law is applicable, so the outgassing rate Q(t) = (dP/dt)(t)V where V is the chamber volume. The system, other than the oil diffusion pump and mechanical fore pump, was entirely constructed of glass.

The test section was 15 mm inside diameter Pyrex tubing divided into two chambers by 15 mm straight bore stopcocks. This air-lock arrangement prevented contamination of the primary measuring chamber during movement of samples into and out of the apparatus. Apiezon L grease was chosen for use on the stopcocks because of its extremely low vapor pressure (10^{-11} - 10^{-13} Torr) at room temperature. The primary measuring chember volume was 138 cm^3 and its internal area was 249 cm^2.

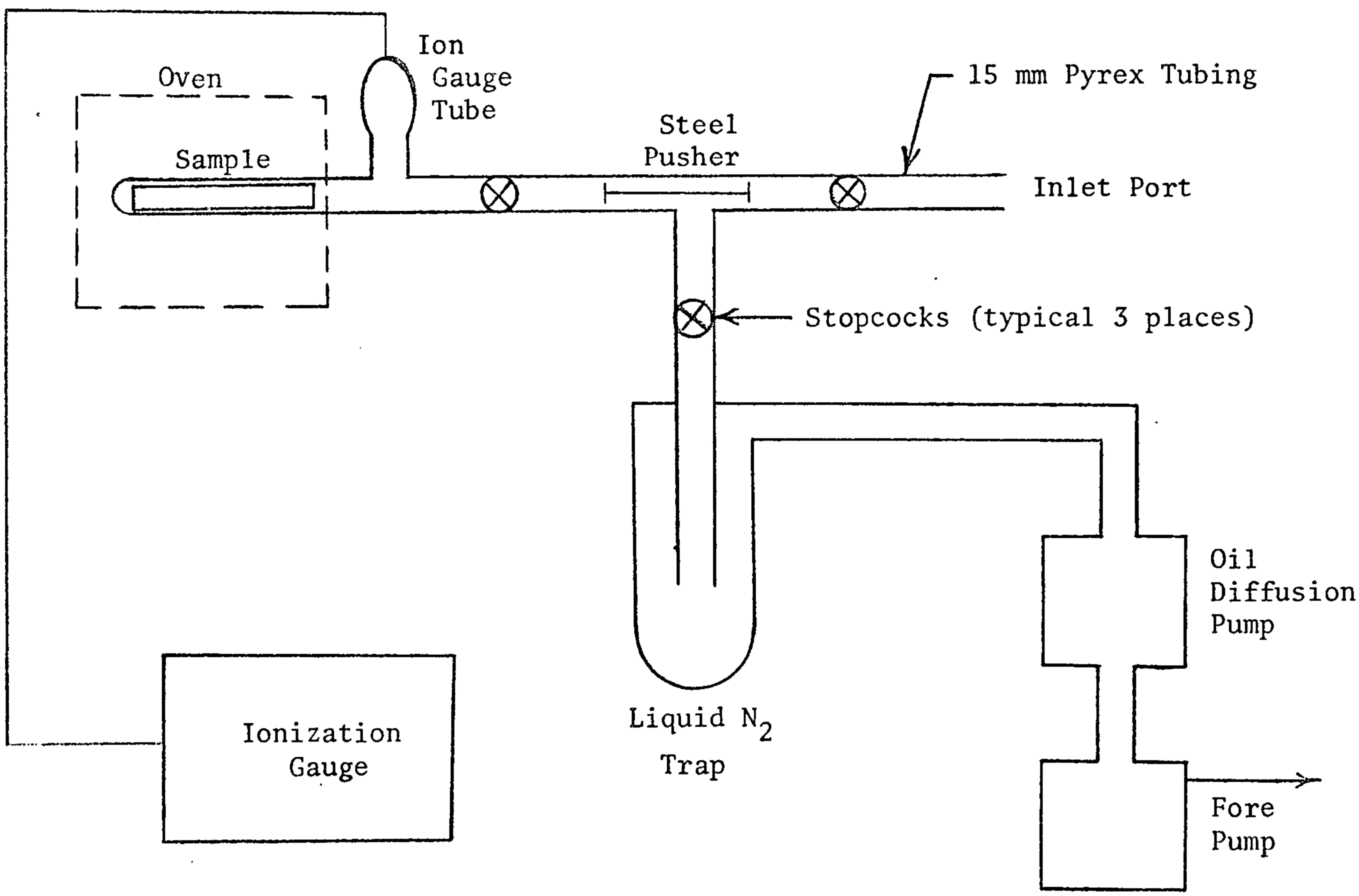

Figure 1. Schematic of Outgassing Apparatus

Samples were manipulated inside the test section by a steel pusher. The steel pusher was moved by an external permanent magnet.

The primary measuring chamber was equipped with a Bayard-Alpert inverted element ionization gauge tube (Cenco No. 94195-2) to measure the pressure. An ionization gauge (Cenco No. 94197) was used in conjunction with the ion gauge tube. This combination was capable of measuring pressure in the range from 1 X 10^{-3} to 2 X 10^{-10} Torr. An auxiliary thermocouple tube and gauge (Cenco Nos. 94179 and 94178-1) were used during initial leak testing of the system. The thermocouple tube was temporarily attached to the sample inlet port and subsequently removed since it was only useful in the high pressure range of 10^{-3} to 1 Torr.

USE OF THE APPARATUS

The first step in using the apparatus was to clean it thoroughly. The glassware was then air baked at near its softening point for several hours. Following air bake, the system was assembled and pumped down. The chambers, ion gauge tube, and trap were given a preliminary vacuum bake for 96 hours at about 600^{o}F. Next, the ion gauge tube and primary chamber were vacuum baked near the glass softening point for 8 hours. Finally, the ion gauge tube was outgassed overnight by turning on its grid and filament currents. This supplied both heat and electron bombardment by the electron emission current.

During vacuum bakeout, the stopcocks were not heated directly in order to prevent losing the high vacuum grease. All three stopcocks were rotated for several hours in order to degas all the grease that would be exposed to the vacuum when the stopcocks were operated. The system was considered degassed when the stopcock isolating the primary chamber could be closed without causing a significant pressure rise in that chamber from the degassing of freshly exposed grease.

The above procedure resulted in a sufficiently low background outgassing rate of the apparatus. The outgassing rate of the aluminum and stainless steel samples was accurately determined by subtracting the background rate from the total outgassing rate. The ultimate pressure attained in this system was 6 X 10^{-8} Torr, when the empty primary chamber was at 350^{o}F.

To insert or remove a sample the primary measuring chamber and the secondary chamber were isolated from the pumping system. Dry air was admitted to the secondary chamber to raise the pressure to atmospheric prior to inserting a sample. The secondary chamber with sample was then pumped down for five minutes before moving the sample to the primary measuring chamber. The pressure at that time was in the 10^{-4} to 10^{-5} Torr range. Pressures observed during outgassing of samples were in the 10^{-5} to 10^{-7} Torr range.

Outgassing rates were measured by isolating the primary measuring chamber and recording rate of pressure rise. It was found that the rate of pressure rise depended to a significant extent on the pressure at which it was measured. This was particularly noticeable at the lowest outgassing rates and lowest pressures. The cause was adsorption pumping of deabsorbed gases by the clean walls of the chamber. To minimize this effect readings were all taken at the same, and conveniently high, pressure of 3.5×10^{-5} Torr.

The background outgassing rate was determined before and after each sample. The before and after values were nearly equal, which demonstrated that the system was well outgassed.

III. DISCUSSION OF RESULTS

The original data of outgassing rate for aluminum and stainless steel samples are tabulated in Table 3. The data are plotted as outgassing rate vs. time for parameters of temperature in Figures 3-6.

To determine the optimum outgassing cycle, it was necessary to determine which cycle removed the greatest volume of adsorbed and absorbed materials. Therefore, Figures 3-6 were graphically integrated to present the volume outgassed as a function of time, temperature, finish, and metal type. The results are tabulated in Table 2 and are plotted in Figures 7-10.

OPTIMUM CYCLE FOR ALUMINUM

The primary methods of outgassing materials are to expose them to a high vacuum for a long period of time and (preferably) at temperatures above 450°F. Unfortunately, some materials are difficult to outgas satisfactorily because elevated temperatures produce unwanted physical property changes, and outgassing proceeds too slowly at room temperature to be practical.

The aluminum tested was a modified 7075 T6. This material has high physical properties which can be reduced by elevated temperatures. Therefore, one portion of this investigation was to determine what temperature-time cycle for aluminum would give the best outgassing with a tolerable loss in physical properties. The allowable time at temperature for 5% reduction or less in room temperature mechanical properties was set by NASA-MSC as a constraint and is reproduced here as Figure 2.

Outgassing rates were measured for aluminum at 285°F, 315°F, and 350°F. At these temperatures, the allowable times (from Figure 2) are 40, 7, and 1 hours respectively. The corresponding total volumes outgassed were read from Figures 8 and 9 and tabulated in Table 1 on the following page. From Table 1, it is apparent that the optimum outgassing cycle lies in the direction of the lower temperatures and associated longer times. Of the temperatures tested, the optimum cycle within the limits of the physical property graph is 40 hours at 285°F.

TABLE 1. Total Volumes Outgassed Within Limits of Graph

Temp (°F)	Limits of Graph (hrs.)	Volume Outgassed (Torr-cm^3/cm^2)			
		7075 Al,32 RMS	7075 Al,63 RMS	304L SS,63 RMS	304L SS,16 RMS
350	1	0.061	0.077	-----	-----
338	2	-----	-----	0.170	0.126
315	7	0.091	0.111	-----	-----
285	40	0.112	0.136	0.335	0.273

Without considering the limitations set by the physical property graph (Figure 2), higher temperatures are strongly favored. The outgassing rates and cumulative volumes outgassed are more than doubled when the temperature is increased from 285°F to 350°F.

OPTIMUM CYCLE FOR STAINLESS STEEL

The room temperature physical properties of Type 304L (ASTM276) stainless steel are little affected by exposure to temperatures in the range of interest here. As shown by Figures 5, 6, 9, and 10, and Tables 2 and 3, the outgassing rates and cumulative volumes outgassed are more than tripled when the temperature is increased from 285°F to 392°F. Therefore, the optimum cycle is the highest temperature and longest time permitted by any other factors which must be considered.

COMPARISON OF ALUMINUM TO STAINLESS STEEL

The initial outgassing rates for aluminum were less than half those for stainless steel, at a given temperature. Furthermore, the advantage of aluminum over stainless steel became more pronounced with time. This result may be partially explained by the fact that stainless steel often contains large amounts of hydrogen and the diffusion coefficient for hydrogen in type 300 stainless steels is quite high (Eschbach et. al, 1963). According to results by Young (1968), aluminum compared better than 304 stainless steel. He did not measure the volume of gas in his samples, nor did he state the type of aluminum. His measurements were taken at 482^{o}F.

EFFECT OF SURFACE FINISH

The 16 RMS and 32 RMS finishes initially outgassed 20% - 35% less than the 63 RMS finishes. The benefit of smooth finish became less noticeable with time, however. At 72 hours the aluminum 32 RMS and 63 RMS finishes exhibited no difference in outgassing rate, while there was less than 10% difference between 16 RMS and 63 RMS stainless steel.

The above result is probably due to the fact that a rough surface furnishes more surface area for adsorbed gases than does a smooth one. The adsorbed gases furnish the main part of the outgassing initially. As outgassing proceeds, desorption of dissolved and occluded gases becomes more significant.

IV. CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

1. For modified 7075T6 aluminum, the optimum outgassing cycle tested was 40 hours at 285°F, when a limit of 5% or less reduction in room temperature mechanical properties was set.

2. Disregarding physical property reduction of aluminum, optimum outgassing was achieved at the highest temperatures and longest times tested, for both aluminum and stainless steel.

3. Aluminum outgassed less than half as much as stainless steel.

4. The smoother finishes gave somewhat lower outgassing rates initially. However, the difference decreased with time and was not appreciable at 72 hours.

RECOMMENDATIONS

1. The above conclusions should be applied, along with other factors not the subject of this study, in establishing outgassing cycles and materials used for the Apollo lunar sample return container.

2. Optimum outgassing cycles should be determined for other materials in the Apollo lunar sample return container. Two components in particular which deserve testing are the cloth bags and Teflon. The cloth bags and Teflon are known to outgas much more than aluminum or stainless steel.

V. REFERENCES

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APPENDIX A

TABLES

TABLE 2. Cumulative Volumes Outgassed from Aluminum and Stainless Steel

Time (hr.)	Volume Outgassed (Torr-cm^3/cm^2) at Temp. (^{o}F)											
	Type 7075 Aluminum						Type 316L Stainless Steel					
	32 RMS			63 RMS			16 RMS			63 RMS		
	285	315	350	285	315	350	285	338	392	285	338	392
0.5	.014	.022	.037	.017	.029	.049	.029	.054	.095	.038	.072	.133
1	.023	.038	.062	.028	.046	.077	.048	.091	.160	.065	.118	.225
2	.033	.055	.088	.042	.068	.107	.071	.127	.230	.092	.168	.321
4	.046	.074	.117	.058	.091	.141	.096	.173	.314	.125	.229	.444
8	.061	.097	.152	.077	.117	.181	.131	.235	.430	.169	.311	.606
16	.080	.125	.195	.099	.147	.227	.179	.323	.592	.227	.422	.825
32	.104	.160	.248	.125	.182	.282	.246	.449	.818	.304	.571	1.120
56	.127	.196	.301	.152	.213	.336	.319	.588	1.081	.386	.734	1.431
72	.139	.213	.327	.164	.228	.362	.355	.654	1.222	.431	.820	1.575

TABLE 3. Outgassing Rates from Aluminum and Stainless Steel

Time (hr.)	Outgassing Rate (Torr-liter/sec-cm^2)(10^{10}) at Temperature (^{o}F)											
	Type 7075 Aluminum						Type 316L Stainless Steel					
	32 RMS			63 RMS			16 RMS			63 RMS		
	285	315	350	285	315	350	285	338	392	285	338	392
0.5	77.6	124	212	95.0	143	286	158	311	529	213	394	741
1	40.1	61.0	94.8	50.2	77.4	118	74.5	131	258	99.0	192	386
2	22.9	36.0	53.7	27.5	42.9	60.9	45.9	84.3	156	59.7	112	219
4	13.7	21.9	32.9	15.7	25.2	45.2	31.7	52.5	103	38.1	71.6	142
8	8.45	12.4	19.0	9.39	14.3	25.4	19.6	36.9	68.2	25.6	47.9	94.5
16	5.59	7.98	11.9	6.44	7.78	13.9	14.9	28.9	49.8	18.1	33.1	64.7
32	3.24	4.85	6.87	4.02	*3.33	*5.13	9.65	18.4	31.0	11.4	22.4	44.2
72	1.85	2.77	4.15	2.07	**	**	6.70	12.3	25.8	7.06	14.2	26.6

* Measured at 48 hours

** No measurements taken at 72 hours

APPENDIX B

FIGURES

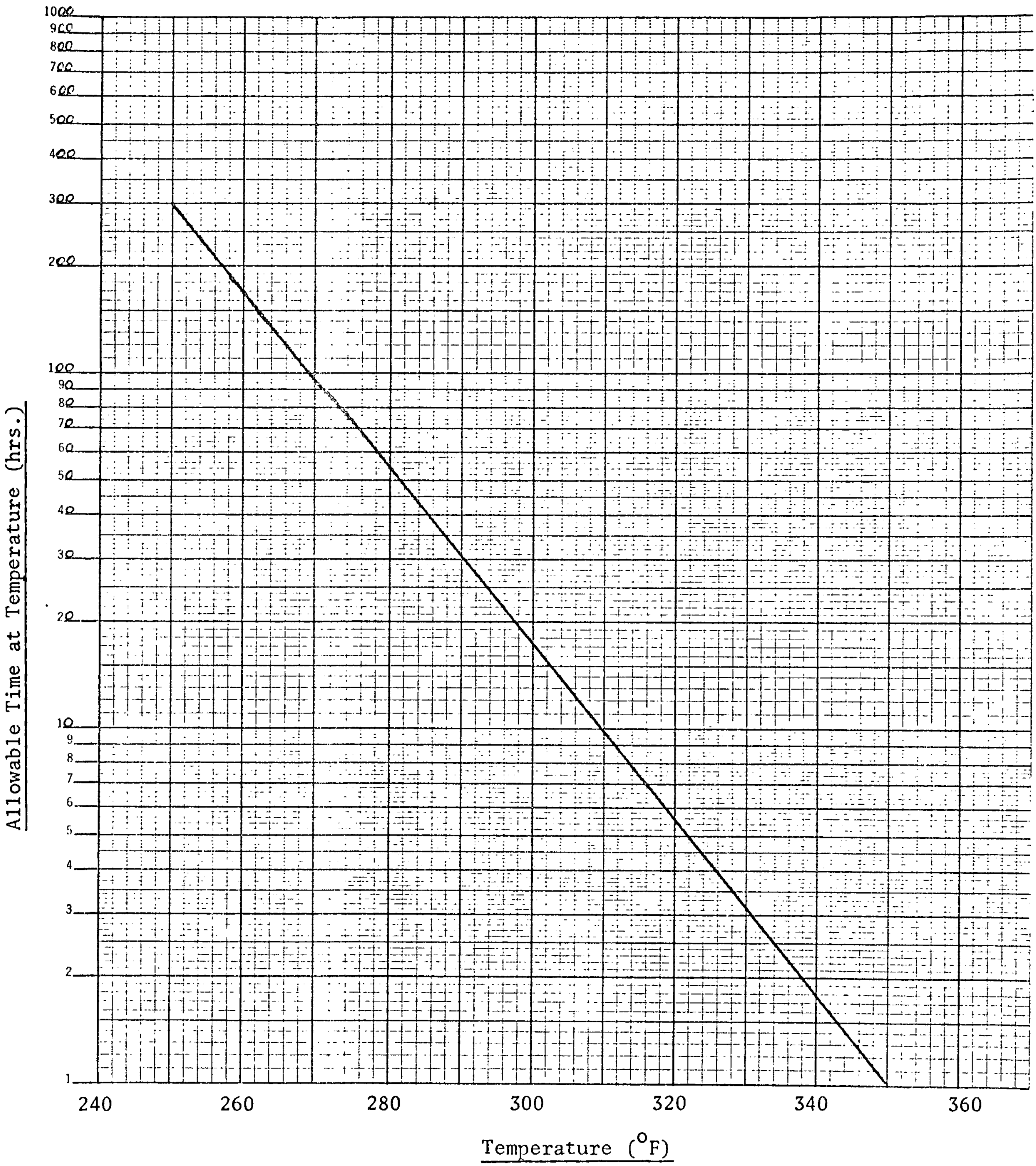

Figure 2. Allowable Time at Temperature for 5% Reduction or Less in Room Temperature Mechanical Properties (Reference: MIL-HDBK-5)

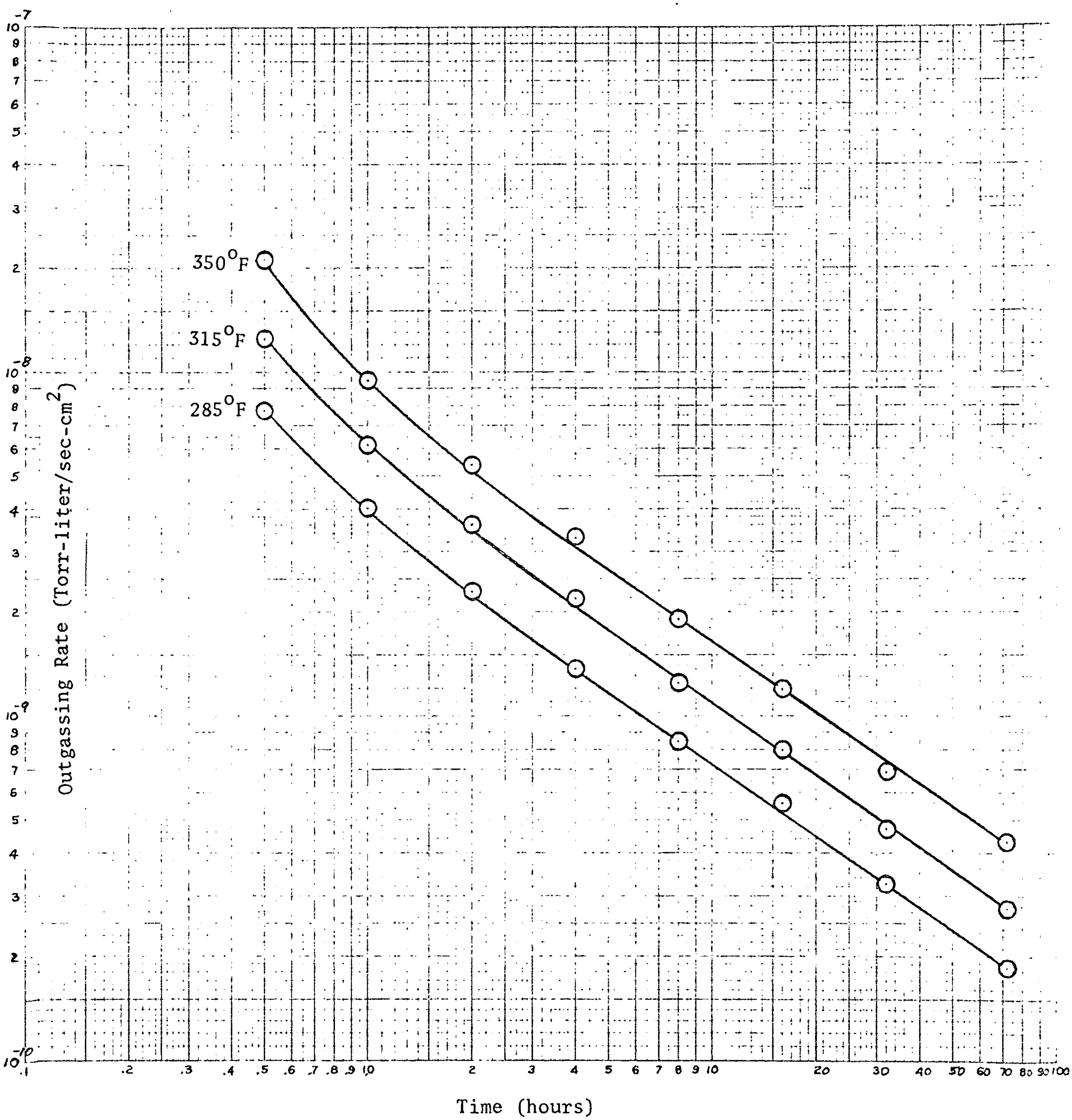

Figure 3. Outgassing Rate vs. Time for 7075 Aluminum, 32 RMS

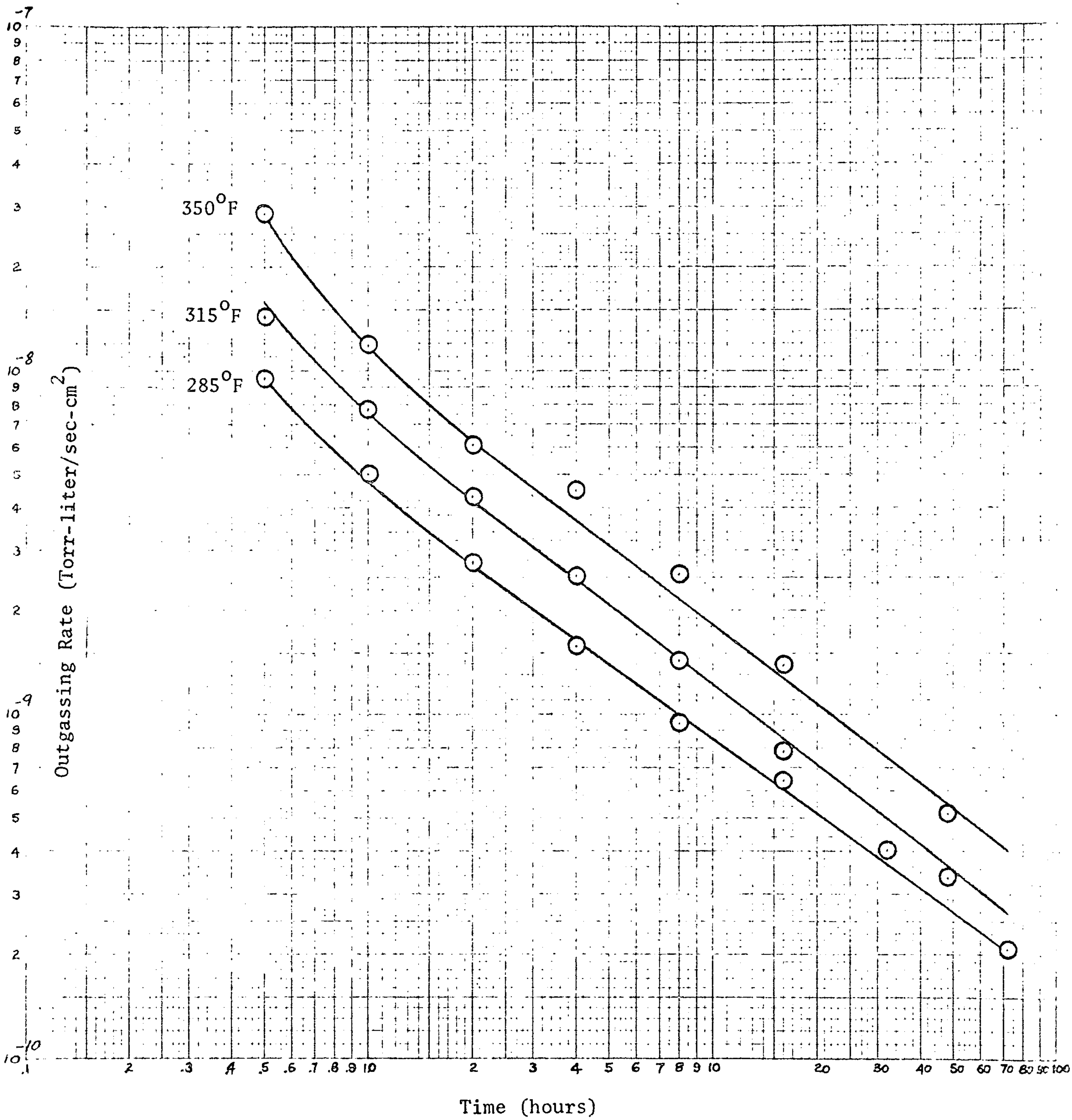

Figure 4. Outgassing Rate vs. Time for 7075 Aluminum, 63 RMS

Full Logarithmic, 3 × 3 Cycles

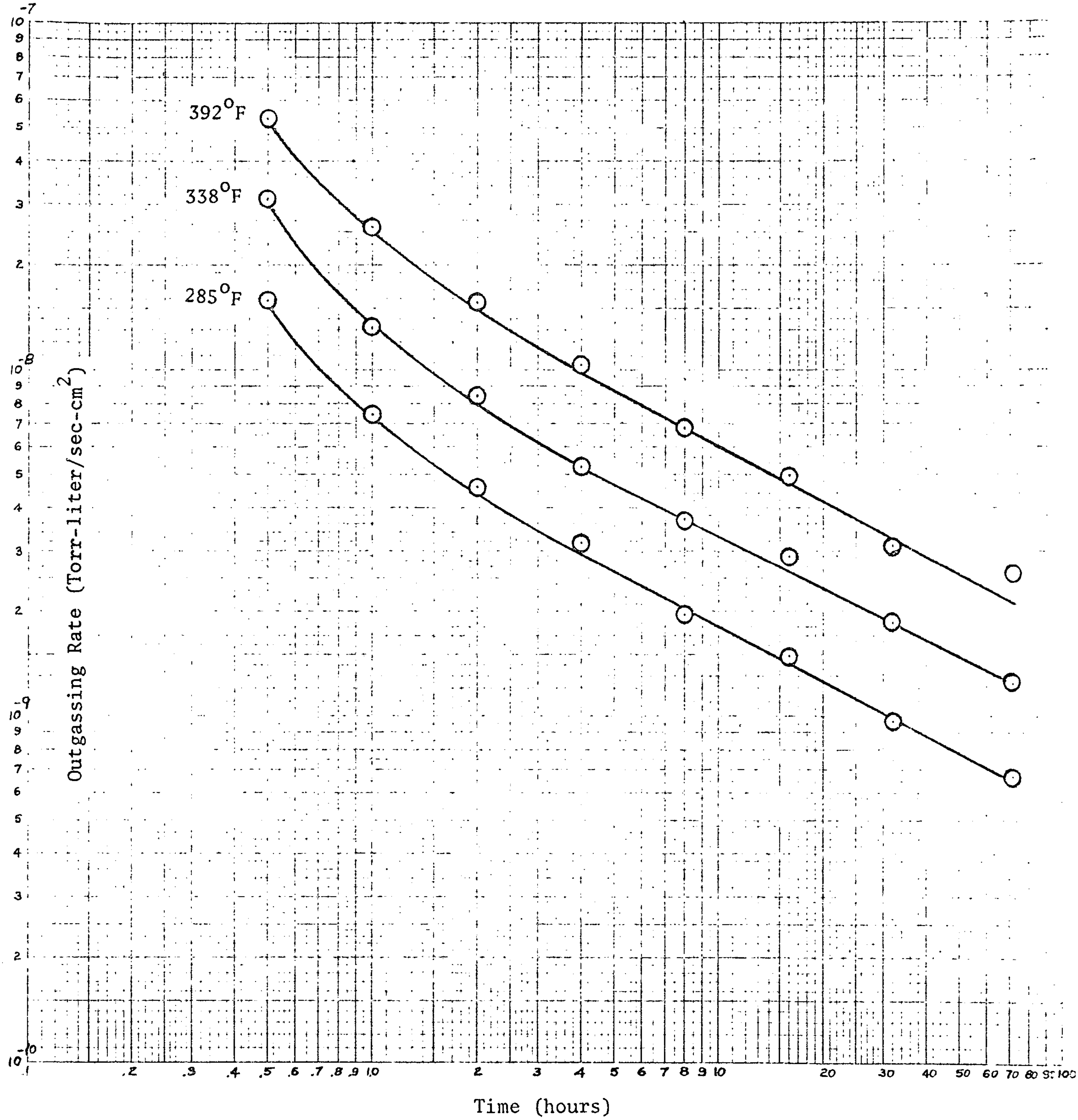

Figure 5. Outgassing Rate vs. Time for Type 304L Stainless Steel, 16 RMS

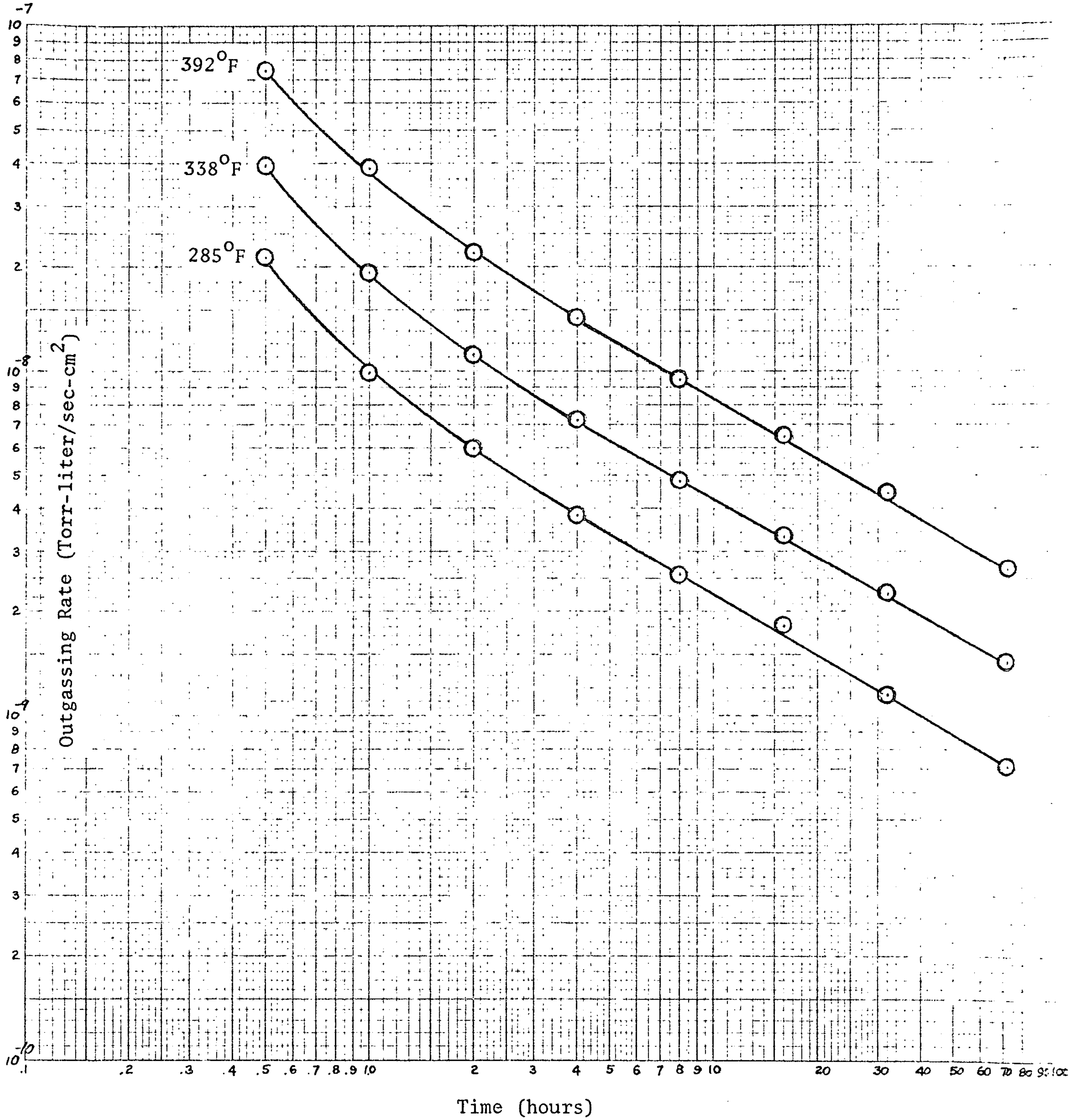

Figure 6. Outgassing Rate vs. Time for Type 304L Stainless Steel, 63 RMS

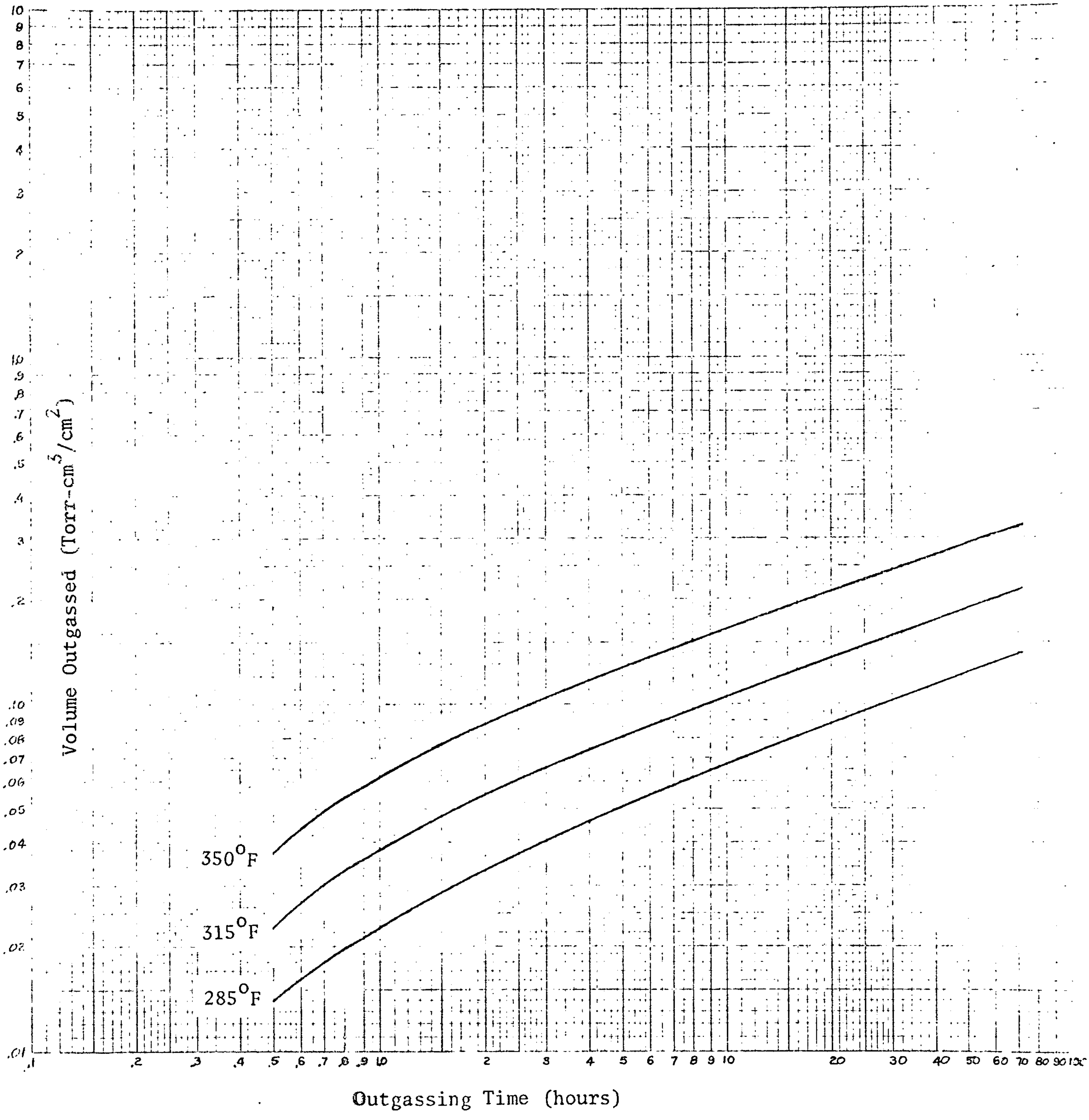

Figure 7. Cumulative Volume Outgassed vs. Time for 7075 Aluminum, 32 RMS

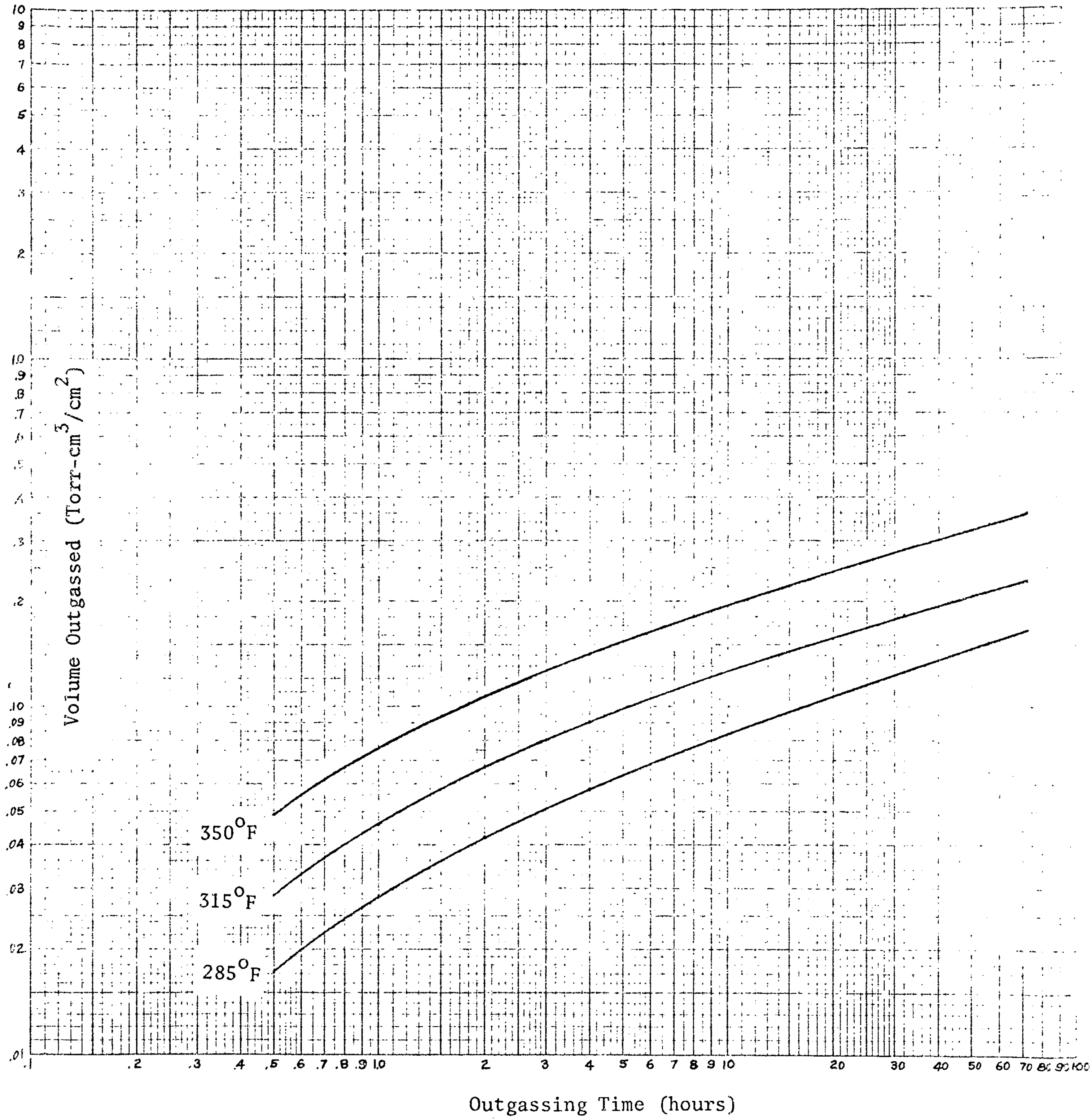

Figure 8. Cumulative Volume Outgassed vs. Time for 7075 Aluminum, 63 RMS

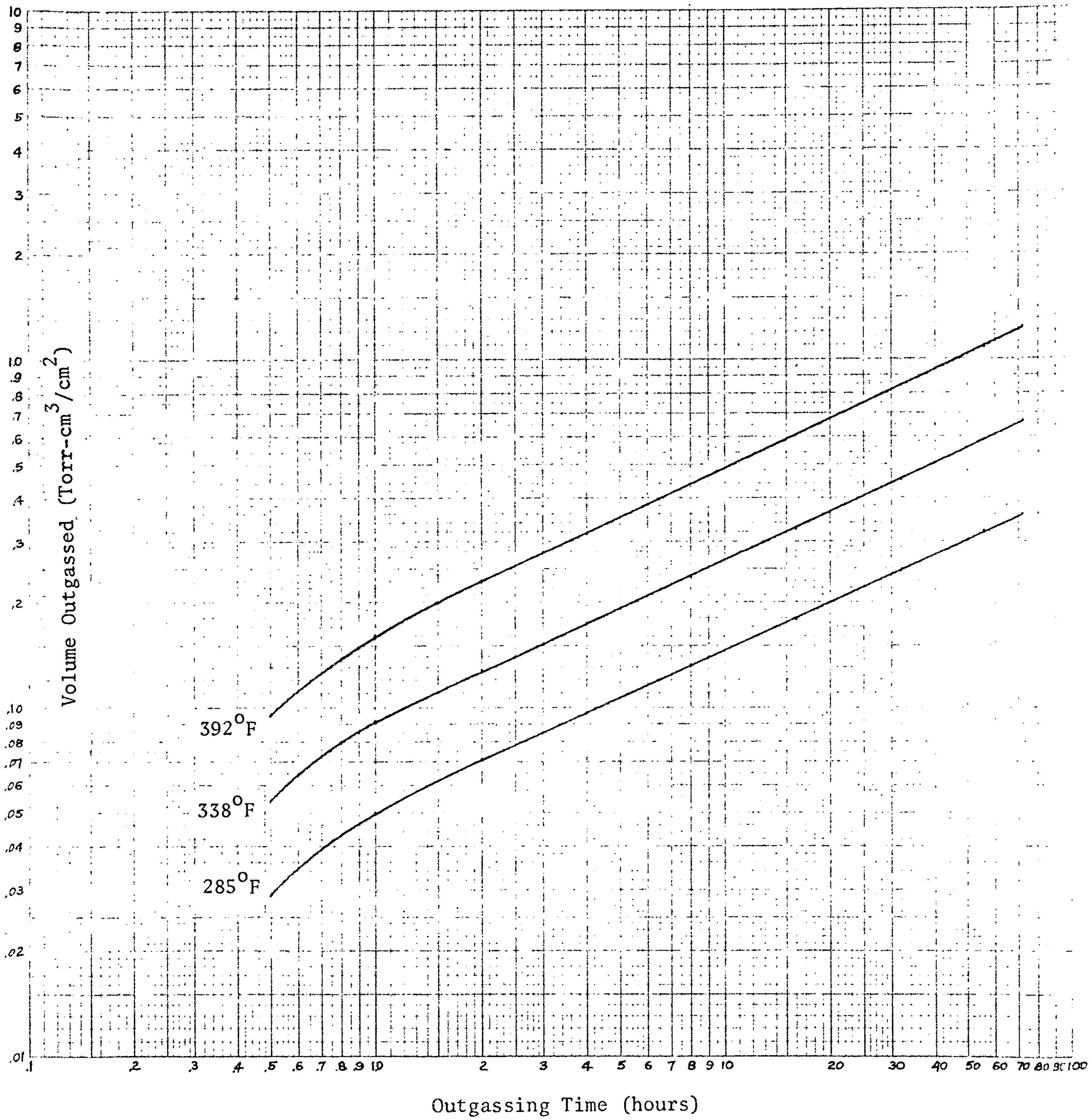

Figure 9. Cumulative Volume Outgassed vs. Time for Type 304L Stainless Steel, 16 RMS

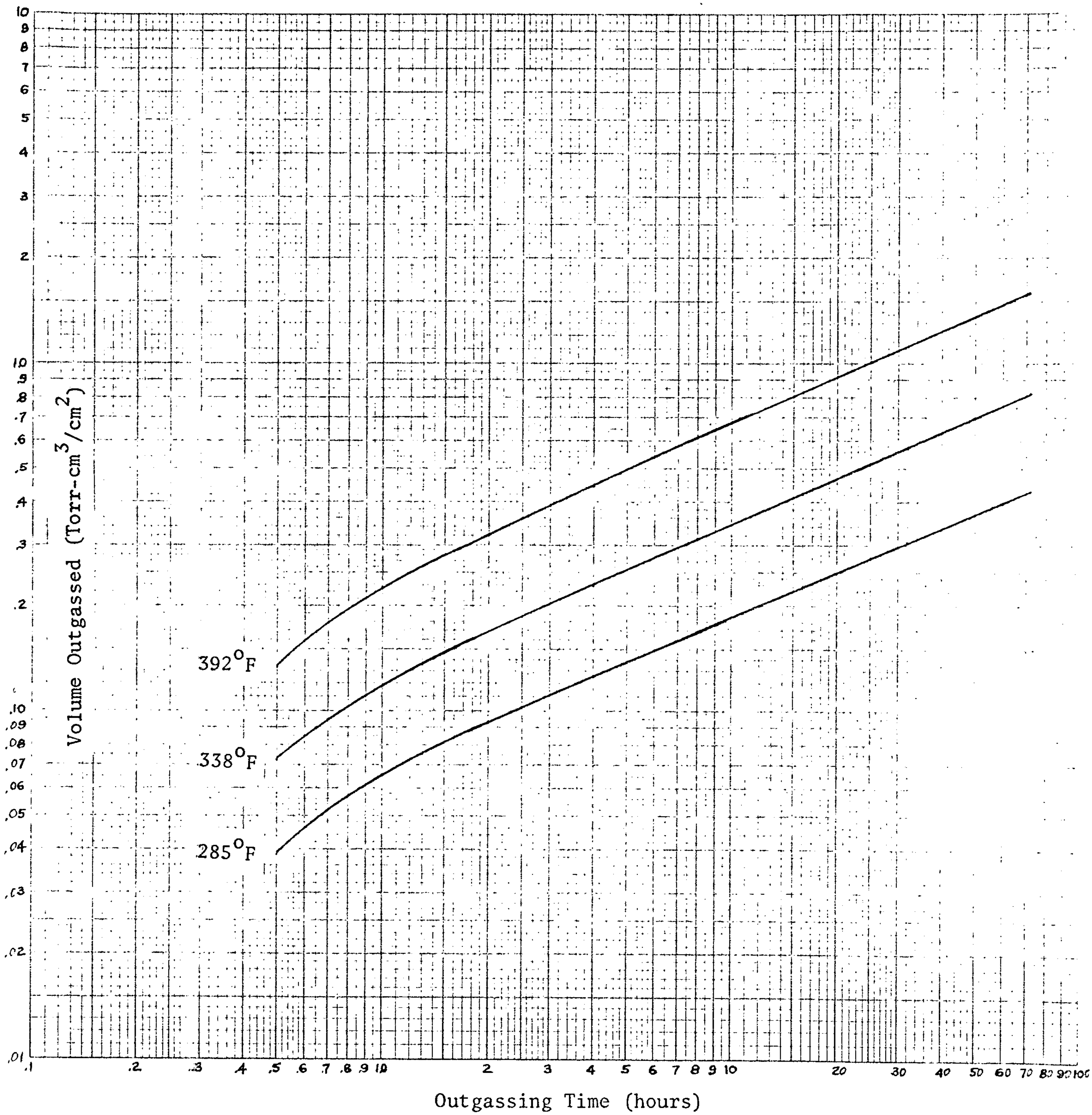

Figure 10. Cumulative Volume Outgassed vs. Time for Type 304L Stainless Steel, 63 RMS