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Teledyne Instrumentation, Inc.
16830 Chestnut Street
City of Industry, California 91748-1020

Report #2 R22A R&D Sensor Testing
Overshoot and Performance

Michael Gonzalez

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INTRODUCTION:

One of Teledyne's distributer (Viamed LTD, UK) has indicated that one of their customers using TAI R22AHJ oxygen sensors has indicated that a high number of sensors tested show upward "overshoot" and unstable readings. The customer noticed the "overshoot" problem occurs when the sample gas is switched from 100% N2 and then to air (20.9% O2/N2). Per customer, the sensors were;

1. Exposed to 100% N2 for approx. 4-6 days.
2. Then, sample gas was switched to air (20.9% O2).
3. Air output readings would "overshoot" for several hours.
4. After this time, air output readings slowly decrease to stable readings.
5. At times when the gas was switch to air, the air readings would be erratic/unstable and
6. Readings would not stable after several hours.

To address these issues, another 20 R22A R&D sensors were assembled with following changes:

1. The R28FT cathode sub-assemblies (B68075) with a thicker 30 micro-inch Rhodium plating were used.
   Note 1: The special B65566 with the special 30 micro-in Rh plating cathodes were delayed. Therefore, to quickly get some test results, B68075 assemblies were used since they already had the 30 micro-in Rh plating thickness.
   Note 2: During the assembly with the R28FT sub-assemblies, defective and double rows cathode assemblies were screened out and eliminated as much as possible (by microscope visual inspection).
2. Ten (10) pcs had the sintered lead assy flushed and then vacuumed to eliminate bubbles/air pockets.
3. Ten (10) pcs did not the flushing or vacuumed de-gassing processes.

So, the purpose of this report is to show that by changing the Rh plating thickness on cathode sub-assy and by adding a couple of new assembly processes listed above, the "overshoot" issue will be greatly minimized and most probably completely eliminated.

PROCEDURE:

Twenty (20) R22A R&D sensors were specially manufactured for over-shooting and stability testing.

1. In the dry sub-assemblies, the R28FT plated cathode sub-assemblies (B68075) were used in place of the standard R22A cathode (B65566).
   - Set #1: 10 pcs had the sintered lead flushed and then vacuumed to eliminate bubbles/air pockets.
   - Set #2: 10 pcs did not have the flushing or vacuumed de-gassing processes performed; they were assembled exactly the same as the standard R22A sensors.
2. For Set #1, each sintered lead assy was thoroughly flushed with E type electrolyte for 15-20 seconds using a large plastic eye-dropper to eliminate any small and loose lead particles.
3. After the expansion membranes were heat-sealed, the wet sub-assemblies were filled with standard "filling" type E electrolyte.
4. Next, the wet sub-assemblies were put inside desiccator container with a vacuum pump tube attached to the inlet port.
5. All sub-assemblies were then "de-gassed" (sometimes referred as "de-bubbled") under a vacuum to eliminate small trapped bubbles/air pockets within sintered lead.
   De-bubbling (de-gassing) - Step by step process:
   a.) The desiccator top was attached to the container and the vacuum pump was started.
   b.) After the 10 minute period, the pump was stopped and the desiccator top removed.

c.) Then, all of the assemblies were re-filled/replenished with more electrolyte solution.
Note: when assemblies are under vacuum, air bubbles escape from within the sintered lead and some of the electrolyte solution tends to splashed out from the assemblies.
d.) After re-filling, the desiccator top was again attached and the vacuum pump re-started again for another additional 3 minutes.

6. After the de-gassing process, the sub-assemblies were re-filled/replenished with more electrolyte solution for final completion for sensing membrane heat-sealing.
7. After all 19 wet assemblies were finished, they were checked in the leak station and then final completed w/pcb assy, diverter cap and contact cap. Note, 1 assy from Set #2 was damaged and not used.
8. After 1 day air stabilization, the output (mV) in ambient air of each sensor was measured (Table 1).
Table 1:

02/11/14	Outputs in ambient air (mV) at 73 deg F (22.8 eg C):						
	Sen	mV			Sen	mV	
	1	10.62	Set #1		11	9.56	Set #2
	2	10.08			12	11.15	
	3	9.37			13	9.86	
	4	9.95			14	9.95	
	5	9.88			15	9.98	
	6	8.88			16	9.98	
	7	10.71			17	10.03	
	8	12.00			18	11.24	
	9	12.27			19	9.69	
	10	12.43			20	NA	
	Avg =	10.62			Avg =	10.16	
	Min =	8.88			Min =	9.56	
	Max =	12.43			Max =	11.24	

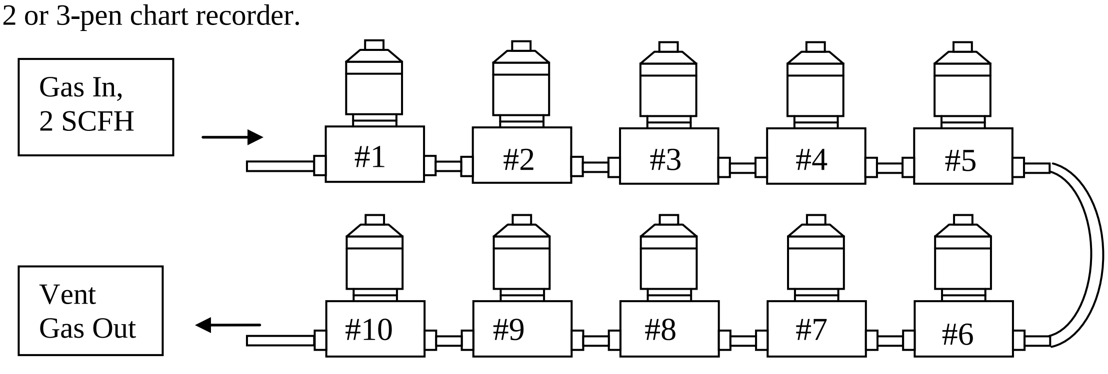
9. After test, the sensors were heat-sealed in barrier bags and stored.

TEST SET-UP AND RESULTS:

Test Set-Up:

A 10 station manifold with flow adaptors set-up was arranged for testing different sample gases.

1. The adaptor blocks were installed in series (see diagram below).
2. A 4-way valve was connected on the inlet of the block #1. The valve would switch between compressed air (20.9% O2) and 100% Nitrogen gas (N2).
3. To recorder/monitor the mV output of each sensor, 10 Molex connector cables were used.
4. The 3-pin female part of the connector was connected to the 3-pin male connector on the back of the each sensor.
5. The opposite end of the connector with a 10 K-ohm resistor across the red and black wires was connected to a 2 or 3-pen chart recorder.



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Test Procedure #1: for testing air over-shooting, air stability and 100% O2 ratio:

1. After 3 day storage period, the 10 sensors one-by-one were removed from their barrier bags and then threaded onto a nylon flow adaptor (compressed air flowing thru @2 scfh).
2. After 15 mins of air exposure, the mV output reading of each sensor was measured (see Table 1).
3. Then, sample gas was switched to N2 gas @2 scfh and test left un-disturbed for approx. 4-6 days.
4. After the 4-6 day period, the sample gas was switched to air @2 scfh for approx. 60 minutes.
5. Next, sample gas was switched to 100% O2 @2 scfh for approx. 15-20 mins (see Table 2 for readings).
6. After testing, the 10 sensors were removed and re-bagged in barrier bags again for storage.

Test #1 Results: Sen #11-19 (without the sintered lead flushing or vacuum de-gassing processes)

	Table 2: Initial Air readings				Table 3: Air and 100% O2 Readings after 4 days in N2 gas.					
02/14/14	**Sen #**	**Air Reading after 15 mins**	To N2	02/18/14	**Sen #**	**after 4 days**	***To air, Any OverShoot?**	**Air Reading after 1 hr**	**after 15 mins**	**Ratio 100%/Air**
	11	9.90	√		11	0.00	No	9.90	46.62	4.71
	12	11.40	√		12	0.00	No	11.40	53.51	4.69
	13	10.10	√		13	0.01	No	10.10	47.56	4.71
	14	10.20	√		14	0.00	No	10.18	48.22	4.74
	15	10.20	√		15	0.00	No	10.10	47.94	4.75
	16	10.20	√		16	0.00	No	10.13	47.93	4.73
	17	10.20	√		17	0.00	No	10.21	48.44	4.74
	18	11.50	√		18	0.01	No	11.36	53.76	4.73
	19	9.90	√		19	0.00	No	9.87	46.77	4.74
	20	na	√		20	na	na	na	na	na
	avg =	**10.40**			**avg =**	**0.00**		**10.36**	**48.97**	**4.73**
					Note: Readings in mV		Ideal Ratio = 100%/20.9% =			4.78

Summary: <u>No overshoot readings</u> in air were seen; output readings were quite stable without any spikes/erratic behavior. The 100% O2/air ratio were quite good ranging from 4.69-4.75.

Test #2 Results: Sen #1-10 (with the added sintered lead flushing and vacuum de-gassing processes)

	Table 4: Initial Air readings				Table 5: Air and 100% O2 Readings after 5 days in N2 gas.					
02/19/14	**Sen #**	**Air Reading after 15 mins**	To N2	02/24/14	**Sen #**	**N2 Reading after 5 days**	***To air, Any OverShoot?**	**Air Reading after 1 hr**	**after 15 mins**	**Ratio 100%/Air**
	1	10.71	√		1	0.01	No	10.78	50.98	4.73
	2	10.17	√		2	0.01	No	10.23	48.43	4.73
	3	9.55	√		3	0.00	No	9.63	45.59	4.74
	4	10.02	√		4	0.00	No	10.11	47.76	4.73
	5	9.98	√		5	0.00	No	10.05	47.60	4.73
	6	9.11	√		6	0.00	No	9.17	43.55	4.75
	7	10.85	√		7	0.01	No	10.86	51.52	4.75
	8	11.58	√		8	0.00	No	11.58	54.80	4.73
	9	12.68	√		9	0.00	No	13.38	58.24	erratic
	10	12.63	√		10	0.01	No	12.73	60.05	4.72
	avg =	**10.73**			**avg =**	**0.00**		**10.85**	**50.85**	**4.73**
					Note: Readings in mV		Ideal Ratio = 100%/20.9% =			4.78

* Sen #9 is suspect/erratic (due to accidental dropping on the ground)

Summary: <u>No overshoot readings</u> in air were seen; output readings were quite stable without any spikes/erratic behavior. The 100% O2/air ratio were quite good ranging from 4.72-4.75.

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Storage Period:
After the 19 sensors were tested, all sensors were stored in heat-sealed barrier bags.

Test Procedure #2: for re-testing air over-shooting, air stability and 100% O2 ratio:

1. After 1 month of storage, the sensors were one-by-one removed from their bags.
2. Sensors were then tested per same steps in Test Procedure #1.

Re-Test #1 Results: Sen #11-19 (without the sintered lead flushing or vacuum de-gassing processes)

	Table 6: Air readings				Table 7: Air and 100% O2 Readings after 4 days in N2 gas.					
03/19/14	**Sen #**	**Air Reading after 15 mins**	To N2	03/28/14	**Sen #**	**N2 Reading after 4 days**	***To air, Any OverShoot?**	**Air Reading after 1 hr**	**after 15 mins**	**Ratio 100%/Air**
	11	9.87	√		11	0.00	No	9.99	47.22	4.73
	12	11.26	√		12	0.00	No	11.35	53.75	4.74
	13	10.04	√		13	0.01	No	10.14	48.06	4.74
	14	10.17	√		14	0.00	No	10.26	48.60	4.74
	15	10.11	√		15	0.00	No	10.19	48.41	4.75
	16	10.12	√		16	0.01	No	10.16	48.07	4.73
	17	10.22	√		17	0.00	No	10.25	48.60	4.74
	18	11.28	√		18	0.01	No	11.28	53.39	4.73
	19	9.87	√		19	0.00	No	9.91	46.98	4.74
	20	na	√		20	na	na	na	na	na
	avg =	**10.33**			**avg =**	**0.00**		**10.39**	**49.23**	**4.74**
					Note: Readings in mV			Ideal Ratio = 100%/20.9% =		4.78

Summary: <u>No overshoot readings</u> in air were seen; output readings were quite stable without any spikes/erratic behavior. The 100% O2/air ratio were quite good ranging from 4.73-4.75.

Re-Test #2 Results: Sen #1-10 (with added sintered lead flushing and vacuum de-gassing processes)

	Table 6: Air readings				Table 9: Air and 100% O2 Readings after 4 days in N2 gas.					
03/28/14	**Sen #**	**Air Reading after 15 mins**	To N2	04/03/14	**Sen #**	**N2 Reading after 5 days**	***To air, Any OverShoot?**	**Air Reading after 1 hr**	**after 15 mins**	**Ratio 100%/Air**
	1	10.74	√		1	0.00	No	10.77	50.99	4.73
	2	10.22	√		2	0.00	No	10.27	48.58	4.73
	3	9.59	√		3	0.00	No	9.72	46.04	4.74
	4	9.98	√		4	0.00	No	10.15	47.95	4.73
	5	9.94	√		5	0.00	No	10.10	47.83	4.74
	6	9.31	√		6	0.00	No	9.31	44.15	4.74
	7	10.91	√		7	0.01	No	10.87	51.55	4.74
	8	11.60	√		8	0.00	No	11.54	54.63	4.74
	9	na	√		9	na	na	na	na	na
	10	12.86	√		10	0.01	No	12.86	60.61	4.71
	avg =	**10.57**			**avg =**	**0.00**		**10.62**	**50.26**	**4.73**
					Note: Readings in mV			Ideal Ratio = 100%/20.9% =		4.78

* Sen #9 not tested.

Summary: <u>No overshoot readings</u> in air were seen; output readings were quite stable without any spikes/erratic behavior. The 100% O2/air ratio were quite good ranging from 4.71-4.74.

Below are charts of the overshoot testing (after 1 month storage period) for 12 of the 18 sensors:
Chart 1: Sen #1, 2 and 3 (04/03/14) – No overshoot seen when gas was switched from N2 gas to Air.

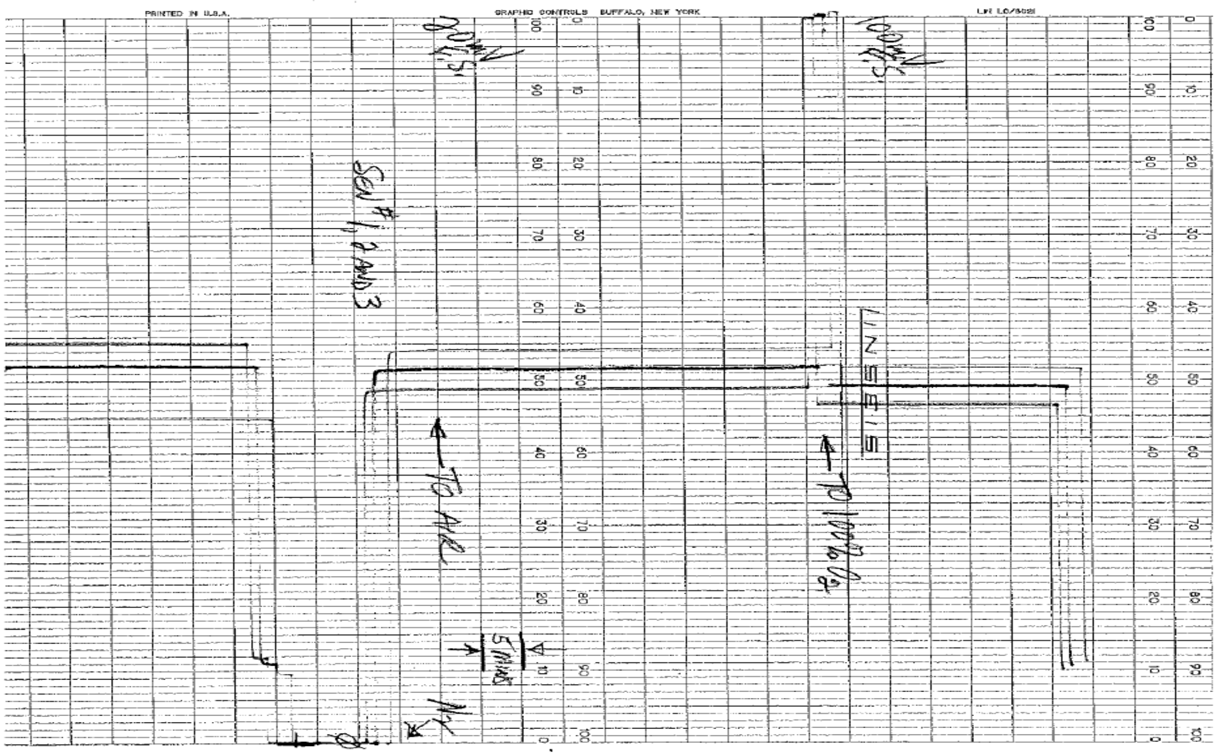

Chart 2: Sen #6, 7 and 8 (04/03/14) – No overshoot seen when gas was switched from N2 gas to Air.

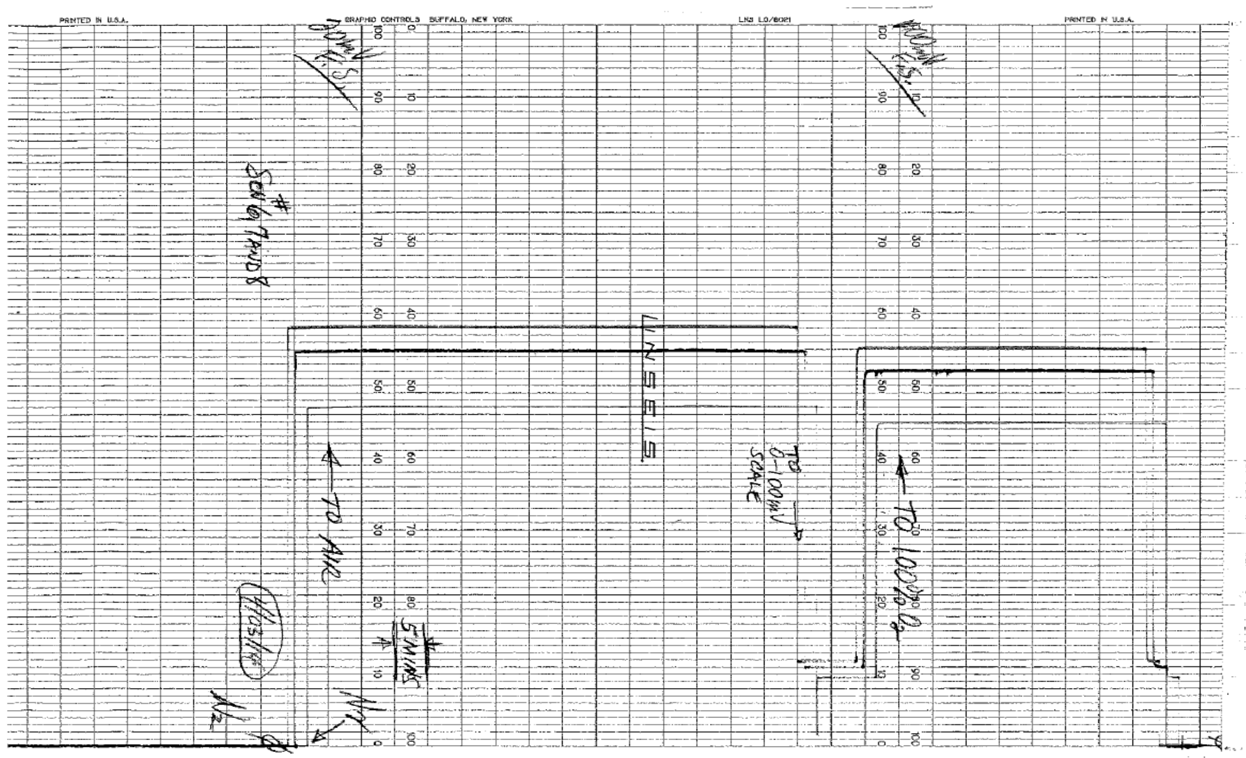

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Chart 3: Sen #11, 12 and 13 (03/28/14) – No overshoot seen when gas was switched from N2 gas to Air.

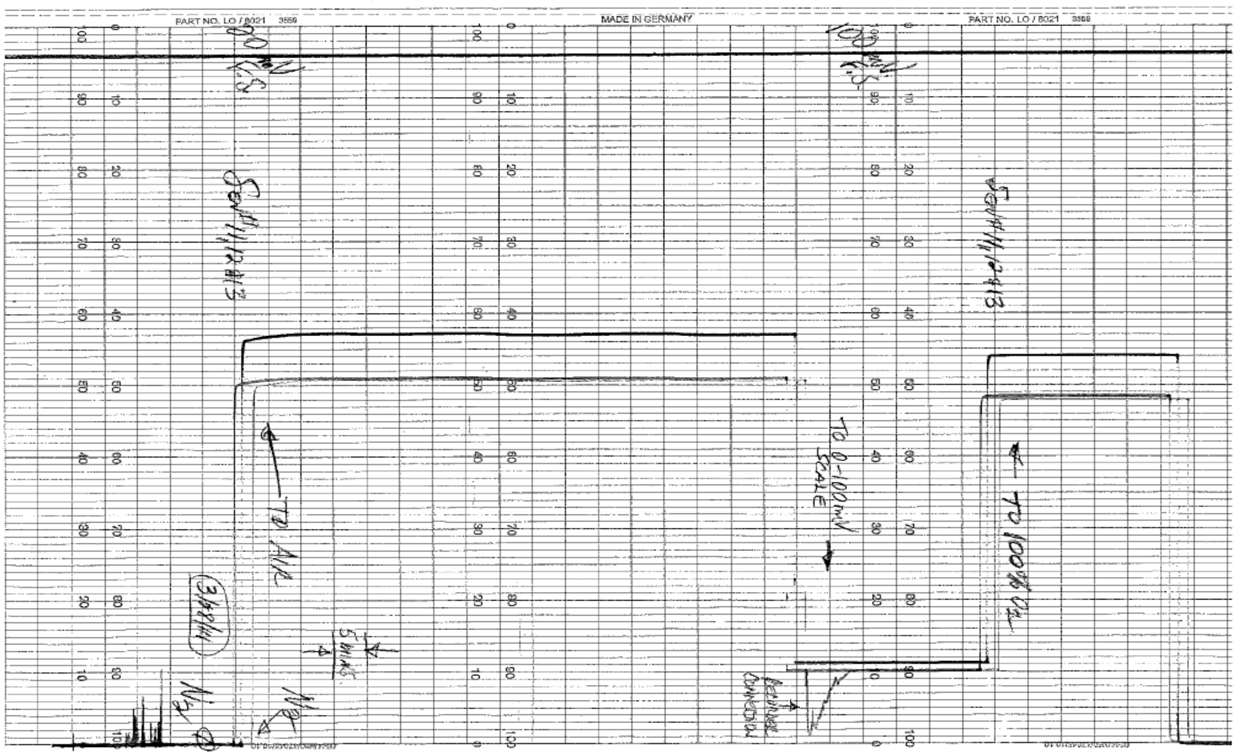
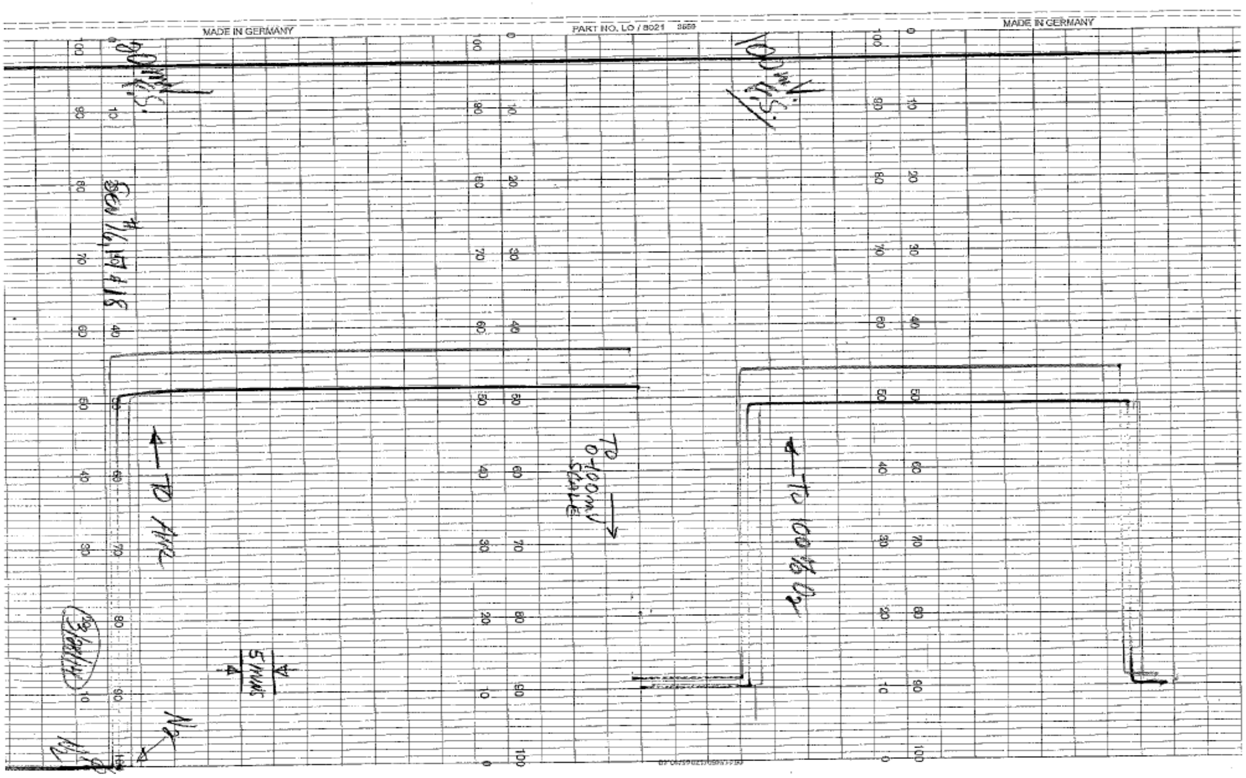


Chart 4: Sen #16, 17 and 18 (03/28/14) – No overshoot seen when gas was switched from N2 gas to Air.



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CONCLUSION:

Twenty (20) R&D R22A sensors were assembled as follows:

1. In the dry sub-assemblies, the R28FT plated cathode sub-assemblies (B68075) with the thicker 30 micro-inch Rhodium plating were used in place of the standard R22A cathode (B65566). Note, during B68075 assembly, defective and double rows are screened out and eliminated by visual inspection.
2. For Set #1 (10 pcs), the sintered lead assemblies were thoroughly flushed with E type electrolyte and then assemblies were then "de-gassed" (sometimes referred as "de-bubbled") under a vacuum to eliminate small trapped bubbles/air pockets within sintered lead. Note: one sensor was scrapped.
3. For Set #2 (10 pcs), the sintered lead assemblies were not flushed or vacuumed/de-gassed; they were assembled exactly the same as the standard R22A sensors. Note: one sensor was suspect.

From the test results presented in this report, all 18 R&D R22A sensors tested in compressed air, 100% N2 and 100% O2 even after 1 month of storage in barrier bags (simulating 1 month of shelf-life storage):

1. No “overshoot” readings in air were seen when the sample gas was switched from 100% N2 to air.
2. Output readings in air were quite stable without any spikes or erratic behavior (when sensors were left in air after the switching process from N2 to air with duration time 60 minutes).
3. Also, outputs in 100% O2 were quite stable for 15-20 minute test duration; no spikes or erratic behavior were seen.
4. More, importantly, there was no difference whether sensors were flushed/de-gassed or not; no “overshooting” was seen from any sensor.

RECOMMENDATION:

With the good results presented in this report, it is recommended another batch of B65566 cathodes assemblies be plating with a thicker 30 micro-inch Rhodium plating instead of the 3 micro-inch plating. Then R22A R&D sensors be manufactured using these cathodes and then assembled the same as the R22A sensors (without the sintered lead flushing or vacuum de-gassing processes).

Next, the sensors should then be tested similar to what was performed on this report for further verification – that the “overshoot” issue will be greatly minimized (or completely eliminated) using the thicker 30 micro-inch Rhodium plated cathodes.