Luxel

Crucible Selection Guide

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Contents

Safety Warning and Disclaimer

Luxel crucibles and crucible liners are designed to be used with RADAK® and OLED furnaces. With the proper crucible and liner combination, Luxel furnaces are extremely robust and versatile sources for a wide variety of thermal evaporation applications. Due to high operational temperatures, it is critical for operators to have appropriate training and adhere to all safety recommendations in this manual. Failure to comply with all safety precautions violates the intended use of the crucibles, and increases the risk of equipment damage and/or personal injury. Follow all guidance in this manual, if you have further questions regarding crucibles or RADAK® furnaces please contact Luxel.

⚠ CAUTION:

This document contains important safety information. Critical safety notes are highlighted in CAUTION boxes.

Luxel Corporation assumes no responsibility for damages resulting from the misuse of their crucibles or RADAK® vacuum furnaces. This document is intended to be used as a guide in the selection of the proper crucible for a given application, however, due to differing system configurations and deposition conditions Luxel cannot guarantee the suitability of a particular crucible for a given application. This guide should be used in conjunction with other sources of information to determine the proper crucible for safe operation in your application.

Crucible Specifications by Furnace Model

Table 1: Crucible specs by furnace model

	ITEM NAME	FEATURES	PART #	USABLE VOL. (CC)	TEMP. LIMIT (°C)
RADAK® I	Crucible, Alumina	Lipped rim	10300-1L	1	1500
	Crucible, Quartz	Flat rim	10300-2	1	1000
	Crucible, PBN	Flat rim	10300-3S	1	1500
	Crucible, Organic	Lipped rim	10300-4	1	450
	Crucible, Arc Coated SS	Lipped rim	10300-5	1	900
	Liner, Tungsten	"Tall" Liner	10301-1T	0.9	1500
	Liner, Molybdenum	"Tall" Liner	10301-2T	0.9	1500
	Liner, Tantalum	"Tall" Liner	10301-3T	0.9	1500
	Liner, Nickel	"Tall" Liner	10301-6T	0.9	1200
	Liner, PBN, Flanged	Flanged top	10301-8F	1	1500
RADAK® II	Crucible, Alumina	Lipped rim	20300-1L	10	1500
	Crucible, Quartz	Flat rim	20300-2	10	1000
	Crucible, PBN	Flat rim	20300-3S	10	1500
	Crucible, Organic	Lipped rim	20300-4	10	460
	Crucible, Arc Coated SS	Reduced aperture w/lip	20300-5L	10	900
	Crucible, Arc Coated SS	Open aperture w/baffle	20300-5C	10	900
	Liner, Tungsten	"Tall" Liner	20301-1T	7.5	1500
	Liner, Molybdenum	"Tall" Liner	20301-2S+	7.5	1500
	Liner, Tantalum	"Tall" Liner	20301-3T	8.5	1500
	Liner, Nickel	"Tall" Liner	20301-6T	7.5	1200
	Liner, PBN	Flat top	20301-8	8	1500
	Liner, PBN, Flanged	Flanged top	20301-8F	8.5	1500
	Liner, Quartz	“Tall” Liner	20301-9	7.5	1000
	Liner, Alumina	“Tall” Liner	20301-10	8	1500
RADAK® III	Crucible, Alumina	Lipped rim	30300-1L	100	1500
	Crucible, Quartz	Flat rim	30300-2	100	1000
	Crucible, PBN*	Flat rim	30300-3S	100	1500
	Crucible, Organic	Lipped rim	30300-4	100	460
	Crucible, Arc Coated SS	Reduced aperture	30300-5A	100	900
	Crucible, Arc Coated SS	Open aperture w/baffle	30300-5C	100	900
	Liner, Tungsten*	"Tall" Liner	30301-1T	75	1500
	Liner, Molybdenum	"Tall" Liner	30301-2TW	60	1500
	Liner, Tantalum	"Tall" Liner	30301-3S+	75	1500
	Liner, Nickel*	"Tall" Liner	30301-6T	75	1200
	Liner, PBN*	Flat top	30301-8	75	1500
	Liner, PBN, Flanged	Flanged top	30301-8F	100	1500

**NON-STOCK ITEM. PRICE ON REQUEST*

Crucible Recommendation by Deposition Material

Please use Table 2 *in addition to other resources* to help you choose the best crucible for your application. This table is based on both Luxel and customer experience, and is continually updated.

Table 2: Crucible and liner recommendations by deposition material

Material	Symbol	Crucible	Liner	Melt °C	100mTorr °C	S / D	NOTES
Aluminum Fluoride	AlF_3	Graphite‡		1040	878	S	
Al Phthalocyanine Chloride	ClAlPc	Organic					
Antimony	Sb	Alumina		631	612	S	☠
Antimony Oxide	Sb_2O_3	Alumina		656	512	S	☠
Arsenic	As	Alumina		814	317	S	O ☠
Arsenic Trisulfide	As_2S_3	Alumina		307	626		
Barium	Ba	Alumina	Ta	727	711		O P ☠
Barium Chloride	$BaCl_2$	Alumina	Ta, Mo	962			
Barium Fluoride	BaF_2	Alumina	Mo	1320	1290	S	
Barium Oxide	BaO	Alumina		1925	1720		⚠
Beryllium	Be	Alumina	W	1284	1377		☠
Beryllium Fluoride	BeF_2	Alumina*		800	690	S	
Bismuth	Bi	Alumina	W, Ta, Mo	271	777		☠
Boron	B	Alumina		2076	2247	S	⚠
Buckminsterfullerene	C_{60}	Alumina					
Cadmium	Cd	Alumina		321	320	S	P ☠
Cadmium Bromide	$CdBr_2$	Alumina*		565	411		
Cadmium Chloride	$CdCl_2$	Alumina*		568	487		
Cadmium Fluoride	CdF_2	VC‡		1110	980		
Cadmium Iodide	CdI_2	Alumina*		390	353		
Cadmium Sulfide	CdS	Alumina		1750	950		
Cobalt Chloride	$CoCl_2$	Alumina*		740	594	D	
Calcium	Ca	Alumina		810	689	S	O ●
Calcium Fluoride	CaF_2	Alumina	Mo, Ta	1418	1446		
Cerium	Ce	Alumina		785	1900		⚠ ●
Cesium	Cs	Quartz		29	207		
Cesium Chloride	CsCl	Alumina	W	645	621		
Cesium Fluoride	CsF	Alumina	W	683	610		
Cesium Iodide	CsI	Alumina		621	610		
Chromium	Cr	Alumina		1857	1550	S	⚠
Chromium Chloride	$CrCl_2$	Alumina*		824	750		
Cobalt	Co	Alumina		1478	1640		⚠
Cobalt Chloride	$CoCl_2$	Quartz*		740	594		

Material	Symbol	Crucible	Liner	Melt °C	100mTorr °C	S / D	NOTES
Copper	Cu	Alumina, PBN, Graphite	Mo	1083	1432		
Copper Bromide	CuBr	Alumina*		488	610		
Copper Chloride	CuCl	Alumina*		430	852		
Copper Oxide	CuO_2	Alumina		1235		S	
Cu Phthalocyanine	CuPC	Organic					
Dysprosium	Dy	Alumina	Ta	1409	1262		
Erbium	Er	Alumina	Ta, W	1522	1332	S	
Europium	Eu	Alumina		822	708	S	O
Gadolinium	Gd	Alumina		1312	1330		
Gallium	Ga	Alumina	PBN	30	1282		
Gallium Arsenide	GaAs	Alumina, Graphite‡	W, Ta	1238			☠
Germanium	Ge	Alumina	PBN	959	1557		⚠
Gold	Au	Alumina	Mo, W	1063	1567		⚠
Holmium	Ho	Alumina	Ta	1470	1340		
Indium	In	Alumina	Mo	157	1082		
Indium Tin Oxide	ITO	Alumina		1800		S	
Iron	Fe	Alumina‡		1535	1647		⚠
Iron Chloride	$FeCl_2$	Iron*‡		677	490	S	
Lanthanum	La	Alumina	Ta, W	887	1927		⚠ O
Lead	Pb	Alumina		328	832		☠
Lead Iodide	PbI_2	Quartz, Alumina		412	403		
Lead Bromide	$PbBr_2$	Alumina*		370	440		
Lead Chloride	$PbCl_2$	Alumina		498	492		
Lead Fluoride	PbF_2	Alumina*		824	669	S	
Lead Oxide	PbO	Quartz, Alumina		885	834		
Lead Sulfide	PbS	Quartz, Alumina		1110	752	S	
Lithium	Li	Alumina, SS	Ta		627		
Lithium Bromide	LiBr	Alumina	Ni	550	641		
Lithium Chloride	LiCl	Alumina	Ni	614	677		
Lithium Fluoride	LiF	Alumina	Ni	845	921		
Lithium Iodide	LiI	Alumina	Mo, W	445	630		
Lutetium	Lu	Alumina		1656	1790		⚠
Magnesium	Mg	Alumina		651	509	S	O
Magnesium Bromide	$MgBr_2$	Alumina	Ni	710	400		
Magnesium Chloride	$MgCl_2$	Alumina	Ni	714	660		
Magnesium Fluoride	MgF_2	Alumina		1263	1270		

Material	Symbol	Crucible	Liner	Melt °C	100mTorr °C	S / D	NOTES
Manganese	Mn	Alumina		1244	1082	S	
Manganese Chloride	$MnCl_2$	Alumina	Inconel‡				
Molybdenum Oxide	MoO_3	Alumina		795	703	S	
Neodymium	Nd	Alumina		1021	1500		⚠
Nickel	Ni	Alumina		1455	1697		⚠
Nickel Chloride	$NiCl_2$	Alumina	Inconel‡	1001	615	S	
Palladium	Pd	Alumina		1555	1647	S	⚠
Phosphorus	P	Alumina		597	222	S	☠ 🔥
Polonium	Po	Quartz		254	380		☠
Potassium	K	Quartz		64	267		🔥
Potassium Bromide	KBr	Alumina		734	681		
Potassium Chloride	KCl	Alumina	Ni	770	720	S	
Potassium Iodide	KI	Alumina		681	638		
Potassium Fluoride	KF	Quartz		857	753		
Praseodymium	Pr	Alumina	Ta	931	1617		⚠
Rubidium	Rb	Quartz		38	2600		⚠ 🔥
Rubidium Bromide	RbBr	Quartz*		680	589		
Rubidium Chloride	RbCl	Quartz		715	682		
Rubidium Fluoride	RbF	Quartz*		775	712		
Samarium	Sm	Alumina		1072	840	S	O
Scandium	Sc	Alumina		1397	1562		⚠ O
Selenium	Se	SS		217	297		☠
Silicon	Si	Alumina	Ta	1410	1817		
Silicon Monoxide	SiO	Alumina	Ta	1702	1125	S	
Silicon Nitride	Si3N4	Alumina	Mo	1900	1415†	S	
Silver	Ag	PBN, Alumina	Mo	961	1162		
Silver Chloride	AgCl	Quartz		455	793		
Silver Bromide	AgBr	Quartz		430	650		
Silver Iodide	AgI	Alumina	Ta	558	696		
Sodium	Na	Quartz		98	357		🔥
Sodium Bromide	NaBr	Quartz		750	701		
Sodium Chloride	NaCl	Quartz		801	753		
Sodium Fluoride	NaF	Alumina		993	916		
Sodium Iodide	NaI	Quartz*		660	660		
Sodium Tri-metaphosphate	$NaPO_3$	Alumina		600	950		C,W
Strontium	Sr	Alumina	Mo, Ta, W	771	627	S	O 🔥
Strontium Fluoride	SrF_3	Alumina		1190	1419		
Sulfur	S	Quartz		597	147	S	P
Tellurium	Te	Alumina	Ta, W	450	433		☠
Terbium	Tb	Alumina		1360	1620		⚠

Material	Symbol	Crucible	Liner	Melt °C	100mTorr °C	S / D	NOTES
Thallium	Tl	Alumina		304	706		☠
Thallium Bromide	TlBr	Quartz		460	350	S	☠
Thallium Chloride	TlCl	Quartz		429	358	S	☠
Thallium Iodide	TlI	Quartz		440	370	S	☠
Thorium Bromide	$ThBr_4$	Alumina	Mo	680	480	S	
Thulium	Tm	Alumina		1545	960	S	
Tin	Sn	Alumina	Ta	232	1412		
Tin Sulfide	SnS	Quartz		880	680	S	
Tin Oxide	SnO_2	Alumina		1630		S	
Tin Phthalocyanine	SnPc	Alumina	Mo				
Titanium	Ti	Alumina*		1668	1742		⚠
Titanium Oxide	TiO	Alumina	W, Mo				
Tris(8-hydroxyquinolinato) aluminium	ALQ3	Organic					
Tungsten Oxide	WO_3	Alumina	Mo	1473	1206	S	
Vanadium Pentoxide	V_2O_5	Quartz		670	900		
Ytterbium	Yb	Alumina	Ta	824	647	S	
Yttrium	Y	Alumina		1477	1832		⚠
Zinc	Zn	Alumina		419	408	S	P
Zinc Selenide	ZnSe	Quartz		1525			
Zinc Sulfide	ZnS	Alumina		1850	1130	S	
Zinc Fluoride	ZnF	Quartz		872	810		

Symbol	Description
💧	Hydroscopic
🔥	Flammable in air
☠	Toxic
O	Oxidizes quickly in air
S	Sublimes
D	Decomposes
P	Poisonous to vacuum systems due to low sticking coefficient
†	A usable deposition rate was reported at the indicated temperature, but the vapor pressure is unknown
‡	See note on depositing molten metals with no liner
*	Based on limited data. Use additional information sources to determine compatibility.
ǂ	Non-stock item, price on request.
C	Source material requires conditioning, heat slowly on first run/meltout
W	Material melt tends to fully wet the crucible, check often for escaped material, consider a liner
⚠	High temperature material that may be deposited with the RADAK® furnace but will require exceeding the warranted temperature limit of 1500C. See section on high temp materials.

***NOTE**: Table 2 is not an exhaustive list: if your material is not listed it does not mean that it cannot be thermally evaporated from a RADAK® furnace. Many other materials are possible to thermally evaporate within the 1500C temperature range of the RADAK®; Luxel suggests performing an online search of published literature to determine the experimental details for the material in questions, including crucible used and temperature of evaporation. You may also contact Luxel for further guidance.*

Depositing Materials Above 1500°C

Disclaimer: The RADAK® furnace is warranted for operation up to 1500°C. However, with a suitable power supply it is possible to evaporate many additional materials at temperatures from 1500-1700°C (e.g. Au, Fe, Pd, etc.). **Be advised that exceeding 1500°C at any time voids the furnace warranty**. Evaporating materials from a RADAK® at temperatures >1500°C should be regarded as experimental, since Luxel cannot guarantee the furnace components, nor the Alumina crucibles which are also only intended for operation up to 1500°C. The chemical compatibility of crucibles may also change at higher temperature, voiding the recommendations in the crucible table. Luxel is not responsible for any damages resulting from operation above 1500°C.

High Temperature Materials & Applications beyond 1500°C: Customers should be aware that **the furnace has been repeatedly tested to 1700°C without signs of damage**, making it possible to evaporate a number of additional materials that exceed the warranted limit of 1500°C. This requires a suitable power supply. Our standard power controllers equipped with the optional 60VAC transformer should provide adequate power for this. Please note you must increase the factory default 1500°C setpoint limitation in the Eurotherm controller (see controller manual for details on this). If you choose to void the warranty in order to evaporate these materials, you may find the following application notes helpful:

Au – We use an alumina crucible with no liner, achieving usable deposition rates from 1550-1600°C. Refer to the note on depositing molten metals with no liner.

B – Limited experimentation with boron has shown that it is possible to achieve very low deposition rates at 1600°C, achieving 0.1Å/s using an alumina crucible with no liner. Temperatures in excess of 1600 appear to degrade or destroy the alumina crucible via chemical reaction. Use caution.

Co – We do not have direct experience evaporating Co from a RADAK®. A usable deposition rate might be achieved around 1640°C. We would suggest using an alumina crucible with no liner. Refer to the note on depositing molten metals with no liner.

Cr – Limited experimentation with evaporating Cr from a RADAK® resulted in a usable deposition rate achieved around 1650°C. We would suggest using an alumina crucible with no liner because this material sublimes though a tungsten liner may be needed for chemical compatibility.

Fe – Our refractory metal liners are chemically incompatible with molten iron at this temperature, so we use an alumina crucible with no liner, achieving usable deposition rates from 1550-1600°C. Refer to the note on depositing molten metals with no liner.

Ge – Ramp temperature up and down slowly at 30C/min, achieving a usable rate around 1500°C.

Pd – We do not have direct experience evaporating Pd from a RADAK®. A usable deposition rate might be achieved around 1647°C. We would suggest using an alumina crucible with no liner. Refer to the note on depositing molten metals with no liner. If you need to load a larger quantity of source material, it may be possible to use a tungsten liner, however we have not tested the liner compatibility so this would be experimental.

Ni – We do not have direct experience evaporating Ni from a RADAK®. A usable deposition rate might be achieved around 1697°C. We would suggest using an alumina crucible with no liner. Refer to the note on depositing molten metals with no liner.

Depositing molten metals with no liner

Molten metal may crack the alumina crucible during cooldown UNLESS you limit the amount of source material to a small puddle on the bottom of the crucible that does not touch the side walls or deplete all of the source material before cooling. This minimizes the stress on the crucible from the difference in thermal expansion rates, but it is critical to check for cracks developing after each cooldown. Alternatively, you can load a larger quantity as long as you completely deplete the source prior to cooldown.

Aluminum Deposition with a RADAK® Furnace

Aluminum deposition from a RADAK® remains a considerable challenge due to the tendency for Aluminum to wet the crucible to the top and crack the crucible on subsequent cooldown. At this time we do not have a recommended solution although we continue to pursue options. Pyrolytic boron nitride (PBN) liners inside of an alumina crucible are one alternative that has been effective but the PBN will eventually fail (hence the crucible/ liner configuration). If you choose to use the PBN liner option, please be mindful of the potential for the PBN to fail. We recommend limiting the PBN to no more than 1300C. In testing, the PBN seems to fail during cool down so we recommend a controlled cool down rate (<25C/min) below 660C. To prevent damage to the furnace we recommend checking the PBN liner between each use for signs of failure and be replaced at the first sign of cracking. If the PBN liner does fail, the furnace will be protected from the Al melt by the crucible but the crucible will not withstand a re-heat.

Luxel offers PBN liners with a flanged top rim which eliminate the issue of wicking, but PBN will eventually fail and must be checked as often as possible. These liners drop into and rest on the installed alumina crucible so inspection is easy (no need to remove the outer can) as long as the top of the furnace can be reached. **The flanged PBN liner is offered for aluminum deposition only with the strong caveat that the PBN will eventually fail**. The safest approach is to run the crucible completely dry each run to avoid cracking. If the PBN fails, the aluminum will weld the PBN into the alumina crucible and you may have to replace the vapor shield.

Crucible and Source Compatibility

Luxel RADAK® and OLED furnaces ship standard with an alumina or quartz crucible installed. Alumina is the default crucible option unless otherwise specified. In addition to these standard crucibles, an expanding selection of specialty crucibles and liners are available to ensure compatibility with the widest possible range of source materials. All crucibles are high purity material and have a precision ground lip to provide a vapor seal with the furnace cover, which prevents contamination of the furnace internals.

The two primary considerations in selecting a crucible are chemical and thermal compatibility. There are a number of resources available on the web for determining crucible compatibility with most common deposition materials. We recommend you use this guide in addition to other information sources.

Thermal / Mechanical Compatibility

Thermal compatibility is a concern with materials that melt during evaporation. Molten materials conform to the shape of the crucible. After the deposition when the melt solidifies, differences in thermal expansion rates can cause the crucible to crack. In the case of melting materials, there are three solutions to avoid crucible cracking. Luxel offers metal liners which can withstand the difference in thermal expansion without cracking. If you desire to run the source material without using a metal liner, one solution is to use a limited amount of source material so that the bottom of the crucible is not covered. A second approach which does not require the metal liner is to ensure all material is evaporated from the crucible prior to cooling.

Figure 1: (Left) The best option for molten metals such as copper is to use a compatible liner inside the crucible, in this case a molybdenum liner is compatible with the source material. (Right) An alternative option is to use a limited quantity of source material so that it does not cover bottom of crucible, this will minimize the chance of cracking the crucible on cooldown.

Importance of Routine Inspection

It is important to routinely inspect the crucible (and liner if applicable) for signs of cracking after each deposition cycle, but particularly when running a crucible / material combination for the first time. Cracking will often be evident after the first run with an incompatible material, which should not damage the furnace since the source material is no longer molten when the cracking occurs. However, furnace damage will result on the subsequent run when the source material melts through the newly formed cracks.

Thermal Incompatibility

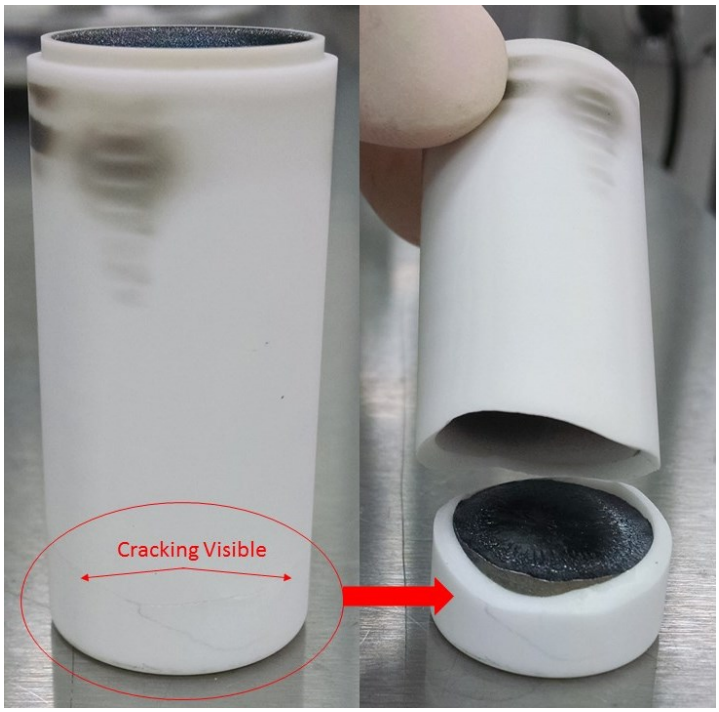

Figure 2: If molten metal wets the entire bottom of the alumina crucible it will likely crack during cooldown.

⚠ CAUTION:

If molten metal wets the entire bottom of the crucible and no liner is used, the difference in thermal expansion can crack the crucible on cooldown. If the operator fails to replace a cracked crucible, then the metal may leak out and destroy the furnace on the subsequent deposition cycle.

Chemical Compatibility

Chemical compatibility also dictates the appropriate crucible material. At high temperatures certain materials may attack, alloy with, or penetrate into the structure of an incompatible crucible, resulting in source material contamination, leakage, and/or crucible failure with subsequent furnace damage. It is important to research the chemical compatibility of your source material and crucible choice to prevent damage as illustrated in Figure 3.

Chemical Incompatibility

Figure 3: Examples of chemically incompatible crucible selections: (Left) Boron in Alumina crucible. (Right) Iron in Tungsten liner.

Crucible Liners

Crucible liners are an insert that may be placed inside of the crucible to achieve the desired thermal / mechanical or chemical compatibility. These applications include source materials that would react with the standard crucible material, and melting materials that have a higher coefficient of thermal expansion than the crucible material. This is especially true of metals, but could apply to any material that exhibits this behavior. Crucible liners contain the molten or reactive material within the standard crucible, preventing damage to the crucible and protecting the furnace.

Crucible liners are available in the following materials:

- Molybdenum
- Tantalum
- Tungsten
- Nickel
- Pyrolytic Boron Nitride (PBN)
- Special materials upon request

Figure 4: A variety of crucible liners are available.

The molybdenum liner is available standard as a full capacity "tall" liner. However, due to the cost of tantalum and tungsten metals these liners are offered in a "short" standard size, having a tradeoff reduction in the usable charge capacity (see Table 1). Therefore, the quantity of material should also be considered when choosing crucible/liner combination. Custom liner sizes are available by special request.

Crucibles

Crucibles are available in the following materials:

- Alumina
- Quartz
- Anodized Aluminum ("Organic" crucible)
- Ceramic-coated Stainless Steel
- Pyrolytic Boron-Nitride (PBN)
- Custom materials upon request

Alumina and Quartz Standard Options

RADAK® furnaces ship standard with either an alumina or quartz crucible installed, with alumina being the default option unless otherwise specified on the order. These high purity crucibles are compatible with a wide range of materials from metals to organic compounds, making them the standard choice for most customers.

Some materials present specific challenges that cannot be overcome using a standard crucible and liner combination. For these applications Luxel has developed specialty crucibles.

Figure 5: Alumina and quartz crucibles.

"Organic" Crucible for Low-Temperature Organics

Organic materials tend to evaporate at relatively low temperatures. Our furnaces are designed to minimizing material buildup at the lip of the crucible by applying more heat to the top of the crucible. Some organics such as ALQ3 and Copper Phthalocyanine are evaporated at such a low temperature that the temperature at the lip of the crucible is insufficient to prevent re-depositing on the crucible. In extreme cases, this can lead to the evaporant choking off the mouth of the crucible. For these applications we have developed a low temperature, high thermal conductivity, ceramic-coated crucible which effectively eliminates crucible choking. This "Organic" crucible is intended for low temperature materials only and should not be used above 460°C.

Figure 6: "Organic" crucible

Ceramic-Coated Stainless Steel Crucible

Originally developed for selenium deposition, our ceramic coated stainless steel crucible is both chemically compatible with the molten selenium and tough enough to withstand expansion during heating and cool down. Lithium may also be deposited from the SS crucible, making this a popular choice for applications in the solar and battery industries. This crucible also features a reduced aperture (on RADAK® II and III sizes) to reduce potentially dangerous source spitting (Figure 7, left).

For materials with a high propensity to spit, Luxel can provide crucible "baffle caps". The caps are installed over the crucible to eliminate line-of-sight between source and substrate. The standard baffle model consists of a stainless steel disc suspended from a stainless cap with an aperture for vapor flux

escape (Figure 7, right). To install a this baffle cap, simply place it on top of an open-aperture crucible (not a reduced-aperture crucible) with the smaller disc hanging down inside, then install the furnace cover over the top of the baffle cap. The furnace cover holds the cap securely in place. Other custom apertures are also possible, including discs with offset perforations to mitigate spitting. We will be happy to discuss your application requirements and design a solution for you.

Figure 7: (Left) Coated stainless steel crucible with reduced aperture to mitigate spitting from Selenium and other source materials. (Right) Crucible "baffle caps" eliminate source-to-substrate line of sight for enhanced protection from spitting.

PBN Crucible

Luxel offers a Pyrolytic Boron Nitride (PBN) crucible. The high purity (99.9% pure) PBN crucibles have a nominal 2% taper over their length. A PBN liner is also available in both straight and flange-top versions.

Custom Crucibles by Request

Luxel can provide custom crucible materials by special request. Our engineering team has a strong history developing solutions for customer applications. Alternative crucible shapes, apertures, materials, and other custom requests should be discussed with a Luxel engineer. Please contact radak@luxel.com or call (360) 378-4137.

Crucible Filling

The appropriate crucible fill level depends on the material being deposited. These can be generalized into three categories: subliming, molten, and chemical compounds.

Subliming Materials

The furnace may be operated with the crucible nearly full of subliming materials in the form of pellets or chunks. During an evaporation at constant power or temperature, the vapor distribution and rate will change with depletion. Few problems have been encountered with materials that sublime or materials with little structural strength, such as most salts.

Non-Subliming / Molten Materials

The maximum recommended crucible fill level for non-subliming materials is ¾ full, resulting in a usable capacity of 1cc, 10cc, and 100cc for RADAK® models I, II, and III respectively (Figure 8). However, special care must be taken when evaporating molten metals: if the residual charge is too large, the alumina crucible may be cracked upon reheating due to the difference in expansion coefficients. Most molten materials require the use of a crucible liner (see Table 2 for the recommended crucible and liner combination by deposition material). The usable volume of a liner will be somewhat less than the nominal charge capacity of the crucible, refer to Table 1 for usable volume details by liner model.

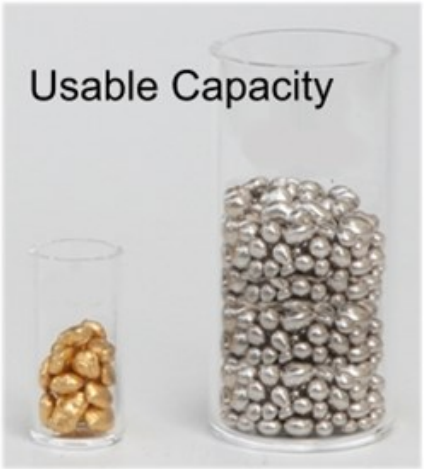

Figure 8: Recommended crucible fill level for non-subliming materials is 3/4 full.

Chemical compounds:

Best results are achieved with the crucible about half-full and depletions down to about one sixth full. Evaporations where vapor qualities such as dissociation, varying rate, or vapor temperature are not a factor can be carried to completion without any other consideration.

⚠ CAUTION:

Failure to use the proper crucible/liner combination with your material may result in leaking molten material into the furnace body, destroying the furnace. For molten materials Luxel recommends inspecting the crucible for signs of cracking after each melt & cool-down cycle.

Crucible Installation Procedure

Always run the furnace with a crucible installed; the crucible is necessary for temperature measurement. To remove or replace the crucible, follow the steps shown in Figure 9: Remove the outer furnace cover by twisting counterclockwise (1) and lifting (2). Center the crucible in the furnace and insert gently until it's supported by the thermocouple (3), or to remove the crucible pull it straight up. Reinstall the furnace cover by aligning with the base and gently pressing down (4), then twist clockwise to engage the locking tines (5). When replacing the cover, check that the crucible is centered with the vapor shield to effect a proper seal against vapors entering the furnace. Lipped crucibles have a lip at the top which locates to the vapor shield and extends slightly beyond it to provide a vapor seal (Figure 10). The crucibles are supported on a spring-loaded thermocouple and the crucible height is critical for making this seal. Crucibles with chipped or broken edges should be discarded.

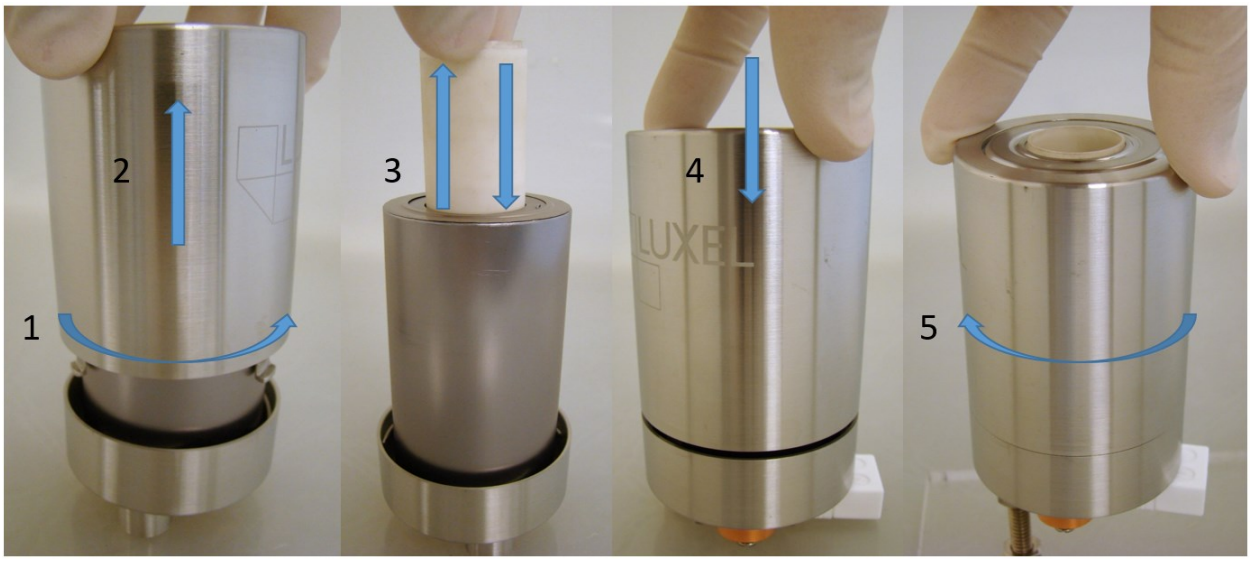

Figure 9: Crucible installation / removal

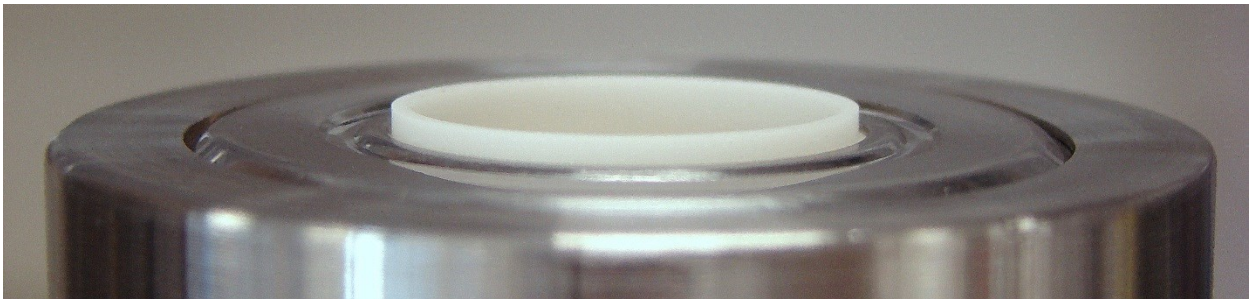

Figure 10: For lipped crucibles, ensure that the lip protrudes from the vapor shield outside the furnace, indicating the crucible is centered and the vapor seal has been properly formed.

⚠ CAUTION:

Make sure that the crucible is centered with the molybdenum vapor shield to effect a proper seal against vapors entering the furnace. Vapors can cause damage to furnace internals.

Cleaning Crucibles

A crucible is typically dedicated to one source material and is not re-used for a different material due to the difficulty of cleaning and the possibility of cross-contamination. However, for some organic compounds it may be possible to clean the crucible sufficiently to re-use it. It can be very difficult to remove organics that have been thoroughly “baked on” to the crucible; success may depend on the particular organic and how hot it was fired. A suggested procedure for removing fired organics is as follows:

- Put the crucible in a beaker of Potassium Hydroxide (0.1M) for 10 minutes
- Rinse with DI water
- Place the crucible in a solution of very hot water and Alconox® Powdered Precision Cleaner (2 tbsp. in 200 ml water) and place it in an ultrasonic cleaner for an hour
- Rinse with DI water
- Place crucible in Acetone and ultrasonic clean for 15 min.
- Place crucible in Isopropyl Alcohol and ultrasonic clean for another 15 min.
- Dry components at 60°C for 30 minutes or longer if required.

This procedure doesn't always render a "new" looking crucible, but it serves well enough to re-use the crucible in some cases. A second approach may be heating the crucible up well beyond the normal evaporation temperature of the organic material and allowing the crucible to “self-clean”. A plasma asher may also work but could take quite a long time. There is always the risk of cross-contamination when re-using a crucible; it is ultimately up to the customer to decide whether it is worth the risk and time to clean a crucible instead of using a new one.

⚠ CAUTION:

In all of these approaches, be watchful for any potential health risks and use proper personal protective equipment/ ventilation.

Removing a Stuck Crucible

In normal operation a crucible should never become stuck in the furnace. Situations that may lead to a crucible sticking in the furnace include:

1. An improper vapor seal between the crucible and the vapor shield (top of the furnace cover)
2. A chemical interaction between incompatible source materials and the crucible
3. A broken crucible which has leaked vapor or molten material into the furnace internals
4. A melted crucible which has been fired too hot

You may attempt to remove a stuck crucible but, depending on the situation, it may or may not be possible to save the heater filament (usually the filament is destroyed since it is relatively brittle). In case (1) above, you can first try dissolving the material in the appropriate solvent if it is a salt or organic compound. If this is not possible, you can resort to mechanical means. Customers have had success removing stuck crucibles by crushing them: try vice grips and/or c-clamps; since these are relatively controllable. Vice grips can be adjusted to close just slightly smaller than the OD of the crucible so you can crush the crucible without damaging the rest of the furnace. A c-clamp affords you similar control. A c-clamp and couple of small nuts strategically placed between the filaments might allow you to salvage the filament as well. The most critical thing to avoid is flexing the heater cage (ceramic and metal structure that supports the filament). Be sure to wear appropriate protection, especially safety glasses.

You may also choose to send the furnace in for repair and Luxel can try to salvage the filament if possible, or install a new heater if necessary.

⚠ CAUTION:

When attempting to remove a stuck crucible by mechanical means, wear appropriate personal protective equipment including safety glasses, and avoid twisting or flexing the heater cage.