Zircoa®

Engineering Guide For Zircoa's Foundry Crucibles

1. Introduction

Thank you for your interest in Zircoa crucibles. The information contained in this Engineering Guide has been compiled over a period of many years, and is based on the experiences of Zircoa's professional team as well as input from many users of our crucible products. The information herein is provided as a courtesy to our customers, and is intended to offer guidance to both novice and experienced founders alike.

Nevertheless, this document is still only a guide. It should not be considered a fixed set of operating instructions. Rather, Zircoa considers it a dynamic document, continuously changing to reflect the latest industry trends and user feedback. Except where safety is a concern, our customers are encouraged to evaluate new ideas and concepts. However, we do ask that results of these evaluations, whether positive or negative, be shared with Zircoa to allow us to continue to improve our products.

The organization of this guide follows a chronological process from crucible selection through analysis of crucible failure modes. Novice users may find it helpful to read through the entire document in the order presented. Experienced users, on the other hand, may be better served by using the guide as an occasional reference or a refresher for specific topics of interest.

Finally, this guide is by no means intended to be the end-all of information and assistance that Zircoa provides to our customers. Our sales and technical staff members are always available to offer support, whether by phone, email or personal visits. Please feel free to contact us to discuss your needs.

2. Choosing the Right Composition

Zircoa specializes in the manufacture of high temperature Zirconia and Zircon crucibles for the investment casting and precious metal industries. This section describes our four basic compositions, and offers some selection guidelines. We also offer application engineering assistance if you are unsure which composition is right for your needs.

Stabilized Zirconia Crucibles

The following three compositions have been specifically developed for use in non-interrupted vacuum melting operations (please see the Data Sheets in Appendix 1 for more detailed information on these compositions):

Composition 3001 is a magnesia-stabilized zirconium oxide with high density and high strength. It is recommended as the "best choice" of crucible materials for melting the entire range of commercial alloys. It exhibits superior resistance to both thermal shock and erosion. Low thermal expansion and non-wettability make this one of our most popular crucibles.

Composition 1651 is a calcia-stabilized zirconium oxide. This composition contains fused grog, and can be used in high temperature (>1800 ^{o}C) applications. It provides superior resistance to erosion, exhibits good thermal shock resistance, and is recommended where high purity is required because of corrosive alloys or high temperatures.

Zircon Crucibles

Composition 2004 is used primarily for melting platinum-group alloys, and is designed for "dirty refining" applications (especially when glassy or siliceous slags are present). Composition 2004 has high strength, is very resistant to cracking, and performs well at very high temperatures. Please see the Data Sheet in Appendix 1 for more detailed information on this composition.

Note: Zircoa offers a variety of crucible backup materials. Please contact your Zircoa representative for recommendations relating to your particular application.

3. Installing Zircoa Crucibles

3.1. Traditional Grouted-Coil Installation (refer to Figure 1)

3.1.1. Be sure that the melt coil is sized appropriately for the selected crucible.

3.1.2. Clean and inspect the coil for damage. Repair areas where grout is loose, thin or missing.

3.1.3. Check the refractory dish (base) of the coil assembly, making sure that it is not cracked or damaged in any way. The dish must be strong enough to support the crucible and its charge at temperature.

3.1.4. Check the new crucible for possible defects: excessive internal pits, holes, cracks, etc.

3.1.5. Be sure to record the crucible batch number.

3.1.6. Place enough crucible backup material (Zircoa composition 1859 recommended) in the bottom of the coil to bring the crucible lip to the desired height (a minimum of ¾" is recommended). Firmly tamp to provide a compacted, level base for the crucible.

3.1.7. Place the crucible into the container and apply a downward pressure while twisting the crucible to seat it firmly in the bottom of the container. Make sure that the space between the coil and crucible is uniform.

3.1.8. Continue adding backup material while ramming it in place with a blunt pointed tool. You may find it convenient to use a small air operated ramming tool, or a hand held vibrator, to help pack the backup material in tightly. When using a ramming tool, be careful not to strike the side of the crucible. When using the hand held vibrator, apply the vibrator to the lip of the crucible as the backup material is poured in. Either method will assist in compacting the backup material.

3.1.9. When ramming is completed (backup material ~1/2" from the top of the coil), pack the remaining space with high temperature wool or rope, or finish with a traditional cap.

3.1.10. If applicable, replace the top seal plate and fasten securely.

3.1.11. Using a vacuum or compressed air, clean out the inside of the crucible and the entire coil assembly.

3.1.12. If the crucible installation was done outside of the furnace, store the entire coil-crucible assembly in a warm, dry place until ready for use.

Note: A properly rammed crucible, supported in compression at temperature, will stay in place through the roughest treatment, and will not leak through to the backup material if minor cracking occurs. Process trials will determine the best technique for each furnace operation.

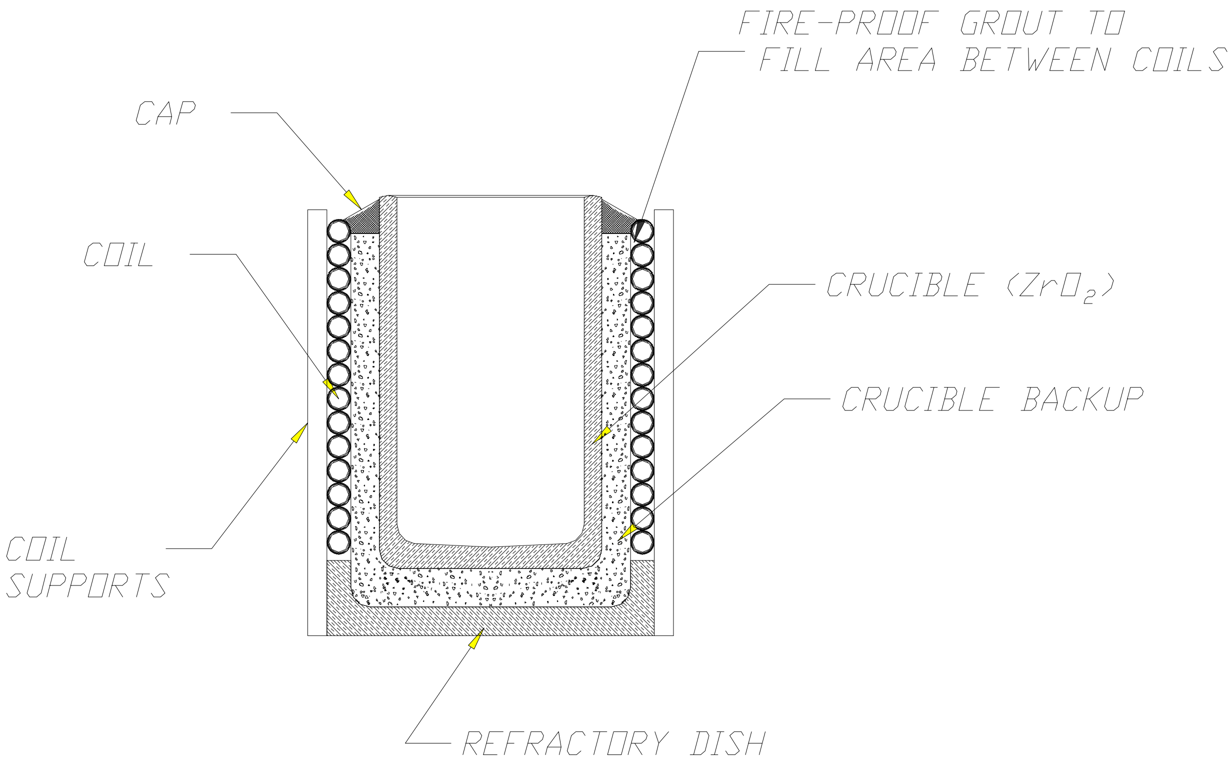


Figure 1. Representation of Traditional Grouted-Coil Installation

3.2. “Two Crucible” Installation (refer to Figure 2)

The “two crucible” or “crucible within crucible” system utilizes a secondary crucible to contain both the melting crucible and the crucible backup material. This concept allows for “pre-installation” of the crucible, at a convenient time and location, therefore eliminating valuable furnace downtime.

3.2.1. Be sure that the melt coil and the backup crucible are sized appropriately for the selected melting crucible.

3.2.2. Check the new melting crucible for possible defects: excessive internal pits, holes, cracks, etc.

3.2.3. Check the new backup crucible for cracks or other defects that might weaken it.

3.2.4. Be sure to record the crucible batch number.

3.2.5. Place enough crucible backup material (Zircoa composition 1859 recommended) in the bottom of the backup crucible coil to bring the crucible lip to the desired height (a minimum of ¾” is recommended). Firmly tamp to provide a compacted, level base for the melting crucible.

3.2.6. Place the melting crucible into the backup crucible and apply a downward pressure while twisting the crucible to seat it firmly. Make sure that the space between the melting crucible and the backup crucible is uniform.

3.2.7. Continue adding backup material while ramming it in place with a blunt pointed tool. You may find it convenient to use a small air operated ramming tool, or a hand held vibrator, to help pack the backup material in tightly. When using a ramming tool, be careful not to strike the side of either crucible. When using the hand held vibrator, apply the vibrator to the lip of the melting crucible as the backup material is poured in. Either method will assist in compacting the backup material.

3.2.8. When ramming is completed (backup material ~1/2” from the top of the coil), pack the remaining space with high temperature wool or rope. Materials that will harden upon drying or use are not recommended.

3.2.9. Using a vacuum or compressed air, clean out the inside of the melting crucible and the “cap” area between the two crucibles.

3.2.10. Store the entire two-crucible assembly in a warm, dry place until ready for use.

3.2.11. Clean and inspect the coil for damage. If grouted, repair areas where grout is loose, thin or missing.

3.2.12. Check the refractory dish (base) of the coil assembly, making sure that it is not cracked or damaged in any way. The dish must be strong enough to support the entire two-crucible assembly and its charge at temperature.

3.2.13. Insert the entire two-crucible assembly into the coil, and secure with clamps or brackets as necessary.

Note: The backup crucible must have sufficient strength under operating conditions to support the expanding zirconia melting crucible without cracking. Although specific backup crucible selection should be based on the needs and testing results of each user, Zircoa composition 6105 (please see Data Sheet in Appendix 1) is recommended as a starting point.

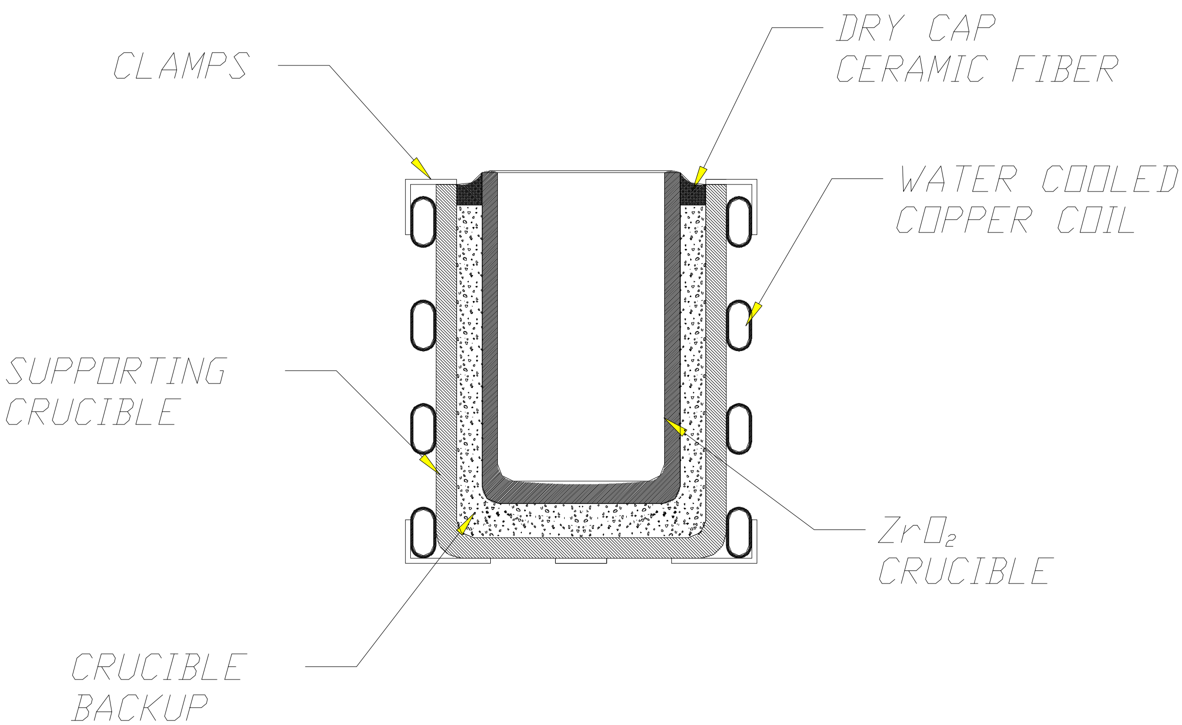


Figure 2. Representation of “Two Crucible” Installation

WARNING!

Note: Zircoa’s crucibles are designed to contain molten materials under a fully supported condition. At no time should Zircoa’s crucibles be used in a free standing or unsupported condition.

4. The Importance of Detailed Documentation

It is important to both Zircoa and our customers that comprehensive records be kept regarding the function and performance of our crucibles. Understanding what factors cause variation in a range of applications and circumstances allows Zircoa to continuously improve its products, and pass these improvements along to our customers.

Therefore, we ask our customers to record, at minimum, the following data for each of our crucibles:

- Crucible lot number (see Figure 3 on following page for an example of our crucible label, along with an explanation of our lot number system)
- Installation method
- Type of backup material used
- Initial heat-up procedure
- Alloy type(s) and weight(s)
- Pouring temperature(s)
- Vacuum level
- Number of pours made
- Detailed reasons for removal from the furnace (shift change, end of campaign, crucible failure, etc.)

Lot ID	Relates to:	Description
05	Batch	Year 2005
03	Batch	3rd month of year (March)
23	Batch	23rd day of month
06	Batch	6th batch of day
357	Kiln	Bickley #357
04	Kiln	4th month of year (April)
01	Kiln	1st day of month
71	Kiln	Car #71

Figure 3. Sample Label and Lot Number Description

5. Using Zircoa Crucibles (Good Foundry Practices)

5.1. General Guidelines

It has been our experience that shop-operating practices substantially influence the performance of our crucibles. A fixed set of operating parameters, which will maximize both crucible life and the quality of castings produced, should be implemented. Both objectives can be accomplished by emphasizing the following and optimizing wherever possible:

- Use consistent and proper installation techniques (see Section 3).
- Avoid damaging the crucible when charging the billets.
- Allow sufficient time for outgassing and uniform preheating on start-up.
- Increase power gradually until normal melting temperatures are reached.
- Pour as cleanly and as quickly as possible to avoid freezing or build-up of metal on the pour area or lip.
- Keep the elapsed time between pours to a minimum.
- Maintain crucible temperature as close to metal pouring temperature as possible at all times.
- Operate vacuum furnaces at the optimal pressure for process and the alloys being cast.
- Develop a melting schedule for each furnace which will allow larger pours to be made first.
- Schedule melting sequence so that “wetting” type alloys will be poured last.
- Schedule melting sequence so that cobalt-based alloys are poured last, and preceded by a nickel base wash melt.
- Schedule frequent visual checks on the condition of the crucible pouring lip and internal surfaces.

5.2. Thermal Expansion Considerations

Figure 3a below shows thermal expansion curves from 0-1300 degrees Celsius for composition 3001 respectively. Due to the nature of zirconium oxide, and its inherent phase changes during heating and cooling, it is important that these curves be considered when using our crucibles.

The blue line on each curve (corresponding to the left y-axis) represents dimensional change, as a percentage, of the material over the temperature range. The red line on each curve (corresponding to the right y-axis) represents the recommended maximum heating rate, in degrees Celsius per minute, of the crucible. It can be seen that a slower heat-up is recommended in the temperature range of about 1100-1300 ^{o}C, where the dimensional changes are most extreme, In general, to avoid problems associated with thermal expansion upon heating, and dimensional hysteresis upon subsequent cooling, Zircoa recommends the following:

- The crucible should never be rapidly heated from room temperature. A moderate heating rate (~15 °C/minute) up to about 1100 °C is acceptable.
- Between about 1100 °C and 1300 °C, a slower heating rate (7-8 °C/minute) is preferred.
- Once the crucible temperature exceeds 1300 °C, every effort should be made to prevent it from cooling below that point.

In addition, Zircoa strongly advises against practices that involve cooling the crucible to room temperature before the end of its useful life with the intent of reheating at some later point in time. As the curves show, the dimensional changes of the crucible during cooling are different than those experienced during heating, which makes it more likely that thermal expansion problems will occur during subsequent re-heating cycles. If an extended period of time between melts is unavoidable, it is recommended that a method of maintaining temperature during this period be used.

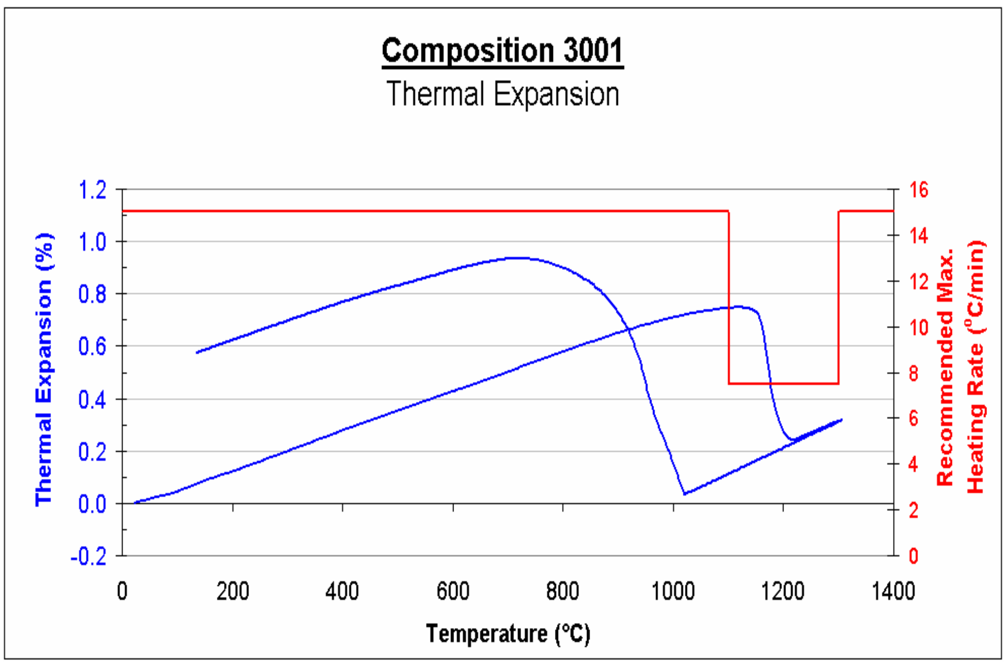


Figure 3a. Thermal expansion curve for Composition 3001.

6. What to Do When Things Go Wrong

6.1. Sharing Information with Zircoa

Despite the best efforts and intentions on the part of both Zircoa and our customers, occasionally problems do occur. When a problem arises, it is in the best interest of both parties to attain a swift resolution. To do so, a thorough evaluation and a high level of cooperation are required.

Therefore, we ask the following of our customers:

- Provide a commitment to Zircoa to purchase a reasonable number of crucibles of one size and composition.
- Agree that no final conclusions regarding performance will be drawn until a statistically significant number of crucibles have been run under a set of fully optimized "fixed operating conditions."
- Arrange to observe the following:
    - Installation and start-up procedures for several crucibles.
    - A statistically significant sampling of melts, noting alloys used, melting temperatures and details of failures.
    - Removal of several crucibles.
- Retain spent crucibles for review.
- Photograph details of concern.
- Report results to Zircoa for review and analysis.
- Allow Zircoa representative(s) to visit your facility to view the problems first-hand.

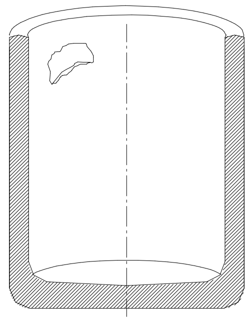
6.2. The "Zircoa Crucible Questionnaire"

Zircoa has developed a comprehensive troubleshooting questionnaire for our customers' use. By completing this questionnaire and sharing the results with Zircoa, we are in a much better position to quickly identify the cause of, and provide corrective action for, any crucible performance problems.

Please contact your Zircoa representative to obtain a copy of this questionnaire.

6.3. Different Types of Failures / Possible Causes

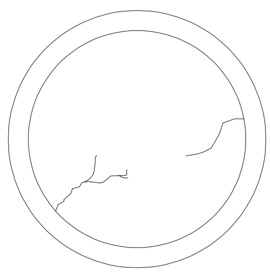
6.3.1. Large “spalls” on the inner sidewalls



Possible causes

- Billet striking crucible wall during charging
- Extreme thermal shock

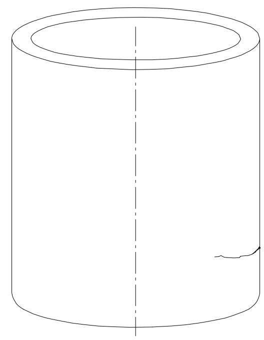
6.3.2. Inside bottom cracks



Possible causes

- Billet striking crucible bottom during charging
- The lack of proper bottom support at temperature
- Crucible allowed to cool excessively between pours
- Incorrect type of backup material
- Not enough backup material

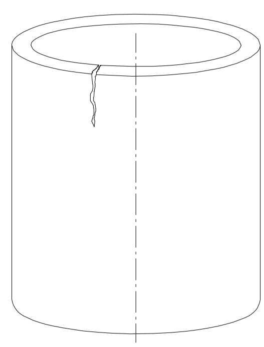
6.3.3. Horizontal cracks



Possible causes

- Non-uniform heating or cooling of the crucible
- Too rapid heating during initial preheat cycle
- Insufficient backup support near bottom of crucible

6.3.4. Vertical cracks



Possible causes

- Lack of adequate or proper backup material
- Ineffective insulation near the top of the crucible
- Excessive thermal cycling of pour lip

6.3.5. Erosion

Erosion is a complex phenomenon, influenced by a variety of factors. Several of the more common forms are discussed below:

6.3.5.1. *Thermal-mechanical erosion* does not involve any interaction between the crucible material and the molten alloy. In this type of erosion, crucible particles dislodge from the crucible body. This is generally caused by either thermal shock or physical abuse, and can usually be prevented by adhering to good, basic foundry practices.

6.3.5.2. *Wetting erosion* occurs with particular molten alloy-crucible combinations with a high interface tension. Although wetting itself cannot be significantly reduced, its effects on the quality of cast products can be minimized by the judicious use of wash melts and proper campaign sequencing.

6.3.5.3. *Chemical erosion* is due to reduction-oxidation reactions occurring between reactive elements in certain alloys and the zirconium oxide crucible. Although impossible to eliminate, the reaction can be reduced by limiting both the amount of superheat and the residence time of the molten alloy in the crucible.