

2700 Curing and Dryout

Abstract

This section tells what must be done to a lining after placement and before firing. The terms “curing” and “dryout” are defined, and curing methods are given for various refractory materials. Then dryout theories and procedures are discussed, including advice on modifying start-up procedures after water washing, hydro-blasting, etc. Typical dryout schedules are also given for five refractory lining systems.

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2710 Definitions

Curing is the initial *ambient* temperature holding period following placement. Not all types of refractory material require a holding period before dryout.

Dryout (also called thermal curing) is the initial heating of refractory linings to temperatures near their service temperature. The dryout procedure forces out any remaining mechanically held water and also expels some chemically combined water. If a controlled dryout is not accomplished, rapid heating may cause explosive spalling or reduce the strength of the refractory. Dryout is completed according to a time/ temperature schedule, and therefore is often done prior to startups of the unit (or other equipment) to achieve better control of the operation.

With the exception of sprayable RCF, most RCF lining systems require neither curing nor dryout.

2720 Curing

2721 Hydraulically Bonded Castables

Hydraulically bonded castables are cured in order to allow time for hydraulic bonding to occur. It is important to prevent moisture loss from exposed surfaces during curing so that an excess of water is available within the refractory to accomplish the various hydrating reactions which develop strength.

Membrane Curing Compound. Curing hydraulically bonded castables (Types A and D through I) is best accomplished by applying a colored (often red) membrane curing compound to the exposed surface. It is important that the compound be applied as soon as practical after placement. This is usually after the material has taken its initial set (about 1 hour). The objective is to minimize loss of moisture from the castable by surface evaporation. Two coats of the curing compound are preferable. This ensures complete coverage. Application is made with a pressure tank garden sprayer. Only resin base curing compounds, such as Master Builder Resin Base, should be used because they are less volatile.

For a minimum of 24 hours after placement, the refractory lining must be maintained between 50°F and 90°F to allow hydration reactions to occur and strength to develop. **Whatever happens, do not allow freshly installed refractory concrete to freeze!** Freezing virtually destroys the hydraulic bonding.

It is highly preferable that curing take place at temperatures greater than 70°F. The reason for this involves highly technical cement chemistry. Basically, we wish to minimize the formation of Al_2O_3 gel during hydration. Better (stronger) castables are achieved when Al_2O_3 gel does not develop in the curing period.

Water Spray Curing. An alternative method (to using a curing compound) is to keep the surface damp with a fine water spray, observing the following provisos:

1. Do not begin water spraying until the material has taken its initial set.
2. Spray the material at 2-hour intervals for a minimum of 24 hours after installation.

Water spray curing is less satisfactory than curing compound. Because of the multiple applications required, water spray curing is usually not done properly.

2722 Other Refractory Types

Type B Refractory. Allow material (such as Resco AA-22) to air dry for 24 hours after installation at a temperature of 50°F to 90°F. Do not water spray or allow water to contact the material. Do not apply a membrane curing compound.

Type C Refractory. Use the same procedure as for Type B (i.e., no compound or water). Some of the erosion-resistant plastics tend to bloat during ambient curing, creating a lamination 1/4 to 1/3 of the thickness below the surface. Fine grained plastics tend to form a skin on the surface restricting water vapor transmission. This tendency is minimized with newer “high-strength,” low-water-content plastics and those with a fairly coarse grain structure. Bloating is definitely undesirable since the delaminations spall off in service, decreasing the lining thickness. An intermediate temperature cure of about 125°F to 150°F for several days has been successful in minimizing bloating.

Regular Plastic Refractory (material that might form the burner wall in a boiler). The curing step should be skipped. If at all possible, the installation of the plastic refractory should be delayed until the boiler or furnace can be fired immediately upon completion of installation. If forms are involved, remove forms as soon as plastic lining has hardened, usually in about 18 hours, and then begin the firing. When it is impossible to fire the boiler right away, cover the plastic refractory wall(s) with polyethylene film, held in place with nails, and keep all manways closed. The objective is to minimize air drying and the formation of drying cracks prior to dryout. Do not allow plastic refractory to freeze prior to dryout.

2723 Two-component Linings

No special considerations are involved when curing a two-component lining. Generally the time required for the back-up layer to set up (harden) and for the hot face anchors to be installed is sufficient curing time before installation of the hot face layer. No special hold times between installation of back-up and working lining are required. Curing compound should be applied to all exposed refractory surfaces, including the back-up layer, within one hour of placement.

2730 Dryout Theories

The mechanism for dryout of a monolithic refractory lining involves applying heat to the hot face and forcing the water to migrate out of the refractory. In early stages of the dryout operation, more water may migrate towards the cooler steel shell than to the hot face. Water that does reach the hot face is removed by evaporation.

As the dryout operation continues, the shell eventually reaches a temperature of about 212°F. At this point, any condensed water begins to vaporize and is forced to move (through small pores and capillaries) through the refractory to the hot surface. Monolithic refractories contain both mechanically held and chemically combined water. In general, the mechanical water is removed first (up to 212°F). As the lining heats up further, above 220°F, the chemically combined water (or part of it, considering the temperature gradient through the thickness of the lining) is also removed.

The objective of the controlled dryout operation is to remove water from the lining in such a way that steam or explosive spalling does not occur. Spalling occurs when water flashes to steam and the resultant increase in volume fractures the refractory. If dryout is too rapid the consequences can be catastrophic. If the lining is cured at temperatures 70-90°F, steam spalling is less apt to occur even when the lining is heated rapidly because of the creation of more permeable pore structures in this temperature range.

Most refractory manufacturers promote the use of various organic fiber additives (see Section 2130) that permit rapid firing (dryout) of installed castable linings. For most types of castables, these organic fibers—which improve capillary structure in the refractory and make it easier for water transport through the lining—may not be required. For thick, dense refractory linings with low permeability and porosity, Types A and D especially, organic fibers do provide a factor of safety during dryout. When used, they lower the strength of the castable slightly. Also, these additives aren't useful in all situations. For example, in an FCC unit, other types of refractory in the unit may limit the heat-up rate anyway.

Currently, several firms specialize in rapid controlled dryouts for new or repaired linings. Their equipment consists of large volume gas fired air heaters and multi-point recorders for monitoring temperature. Chevron has used these firms successfully on a number of jobs. Two companies that do this work are:

1. Hot Work, Inc. – (606) 276-1570
2. Cooperheat – (908) 981-0800

2740 Dryout Procedures

Dryout procedures that refractory manufacturers propose for monolithic refractory linings are generally too conservative. The manufacturers are concerned that the linings may be damaged during dryout by too rapid heating and that they will be blamed for supplying inferior refractory products. The Company's recommended rates are more practical and have proven safe when used with its refractory specifications.

2741 Refractory Castable Linings

Dryout of refractory castable linings means heating the lining at a controlled rate to a maximum temperature. The schedule may include one or more holding periods, and the heat-up rate may increase as well.

Low-temperature Hold. The Company recommends a low-temperature hold (250 to 300°F) for Types E and F semi-insulating castables. For Types A and D erosion resistant castables the Company recommends a low-temperature hold at 325 to 375°F. [52, 63] These temperatures are hot gas temperatures and correspond to about 225°F refractory hot face temperature. In either case the hold time should equal one hour per inch of refractory thickness. The low-temperature hold must be used in all refractory dryout procedures and is particularly important in fired heaters and boilers, because furnace heatups are hard to control, with temperature in different parts of firebox varying widely.

High-temperature Hold. The Company also recommends a holding period at 925 to 1000°F for one hour for types E and F semi-insulating castables. For Types A and D, erosion resistant castables, the Company recommends a three hour hold at 1000°F. [62]

2742 Dryout of Type C Plastic Refractory

Although plastics have a much lower water content than hydraulically bonded castables, they go through some five phase changes before they reach the final reaction at 932°F. They can be mechanically damaged until they reach about 230°F, and they can rehydrate anytime before they reach 932°F. For this reason, the Company recommends that in the field, where several types of refractory material are to be used, Type C refractory work be done last (to prevent mechanical damage). For shop-lined equipment, the Company recommends dryout (above 932°F) before it goes to the field in case the lining should get wet before the equipment is installed.

Typical dryout schedules for monolithic refractory linings are shown in Figure 2700-1.

2743 Dryout of Two-component Linings

When preparing a dryout schedule for a two-component lining (e.g., 3 inches of dense 94% Al_2O_3 castable backed by 3 inches of insulating castable) the first thing to calculate is whether or not the proposed schedule will remove all of the mechanically held water from the back-up castable. Thermal conductivity calculations can be used for this purpose. The lining should be held at the calculated temperature long enough for the lining to reach equilibrium and the cold face temperature to reach 230°F. Monitoring the cold face temperature is an additional way to monitor dryout progress.

The other consideration is the heating rate to be used during dryout. Again, we must control the heating rate to protect the refractory most apt to be damaged by too rapid heat-up, in this case, the dense 90% Al_2O_3 castable. Too rapid heating rates cause explosive spalling. (See Figure 2700-1 for a typical schedule.)

Fig. 2700-1 Typical Dryout Schedules⁽¹⁾

Parameters	5" Regenerator Lining, FCCU ⁽²⁾	5" Vibration Cast Lining, Reactor Riser ⁽³⁾	1" Cyclone Lining	1" Cyclone Lining	Two-component Lining 6-8" thick
Monolithic refractory type (per GB-N1268)	"One-shot," semi-insulating castable, Type E or F	Erosion-resistant castable, Type A or D	Chemical setting erosion-resistant castable, Type B	Chemical setting erosion-resistant plastic, Type C	Typically 3-4" of 94% Al ₂ O ₃ (Type A) and 3-4" insulating (Type I)
Low-temp. Rate/Hold ⁽⁴⁾	Heat at 75°F/hr.; Hold at 250-300°F	Heat at 50°F/hr.; Hold at 325-375°F	Heat at 50°F/hr.; Hold at 250-300°F	Heat at 50°F/hr.; Hold at 250-300°F	Heat at 75°F/hr.; Hold at 250-300°F
High-Temp. Rate/Hold	Continue at 75°F/hr.; Hold for 1 hr.; 925-1000°F; Ready to operate	Continue at 50°F/hr.; Hold 3 hrs. ⁽⁵⁾ at 1000°F; Ready to operate	Raise rate to 100°F/hr.; Hold at 650-700°F; Ready to operate	Continue at 50°F/hr.; Hold at 650-700°F; Continue heat-up to 935°F minimum; Ready to operate	Continue at 75°F/hr. until cold face reaches 230°F; Ready to operate
Remarks	Dryout of lining is usually done during start-up	Install TC's for shop dryout on refractory surface, & use to monitor dryout		Surface TC's for shop dryout should be used to make certain refractory reaches 935°F	

(1) See text. Temperatures are "hot gas" temperatures with some exceptions. These schedules assume no credit for addition of organic fibers.

(2) Refractory Types F, G, H and I follow approximately the same schedule.

(3) Refractory Types D2 and D3 follow approximately the same schedule.

(4) Holding time equals 1 hour per inch of thickness in all cases.

(5) If lining is being dried out by operations, eliminate hold and continue at 50°F/hr. to operating temperature.

2744 Monitoring Dryout Temperature

During dryout of a lining, it is desirable to measure the temperature of the refractory hot face as well as the temperature of the hot gas from the burners. It is preferable that any temperature control (manual or automatic) of the dryout operation be based on the hot-face temperature of the installed refractory rather than on the temperature of the hot gas stream. There are two good reasons for this statement:

- The hot gas stream is typically hotter than the hot face of the refractory.
- If damage should occur to the refractory lining by too rapid heating, the damage will be directly related to *refractory* temperature and not to the temperature of the hot gas stream.

Unfortunately, it is often impractical to place thermocouples on the hot face of the refractory. Also, dryout schedules proposed by the refractory manufacturers are based on hot gas temperatures. For this reason, hot gas temperatures are given in Figure 2700-1.

These schedules have a factor of conservatism built in and should be used to monitor dryout of refractories when a process vessel or a fired heater is brought up to operating temperature after a shutdown.

In the case of an FCC unit, there will be several repair/replacement areas involving three or four different types of refractory. It is not feasible to have thermocouples on the hot face of each refractory location. In this situation, use the slowest schedule of those recommended for the refractory types involved (thickest, densest refractory).

2745 Shop Dryout

It is important that dryout of shop fabricated equipment be accomplished before shipment by heating in a fashion similar to how it is heated in service. Cyclones can be heated from both the inside and the outside in an oven since they are hot wall units. Transfer lines, vessels and other cold wall units should only be heated from the hot face. If the cold (insulated) face is heated in an oven, the following may occur:

1. The shell may be heated above its (metal) temperature limit.
2. Excessive thermal expansion of the shell may occur, causing anchors and/or the shell to break loose from the refractory.

In the shop, it is usually feasible to place thermocouples on the hot surfaces of the linings. These thermocouples should be used to control the dryout operation. A suitable "thermal curing" burner with adequate heat release is essential. In all cases, air flow is extremely important. A cover plate, or damper, should be installed at the equipment or vessel outlet for adjustment of hot flue gas flow.

2746 Field Dryout

It is very difficult to meet the dryout schedule and to hold temperatures constant under field conditions. As an example, if you are drying out a fired heater by using an existing burner(s), the temperature gradient throughout the heater may be significant. In cases like this it may be better and less time consuming to contract the dryout work to a firm which has the proper equipment. If practical, thermocouples should be installed and connected to a field readout monitor after the refractory is installed. These thermocouples must be tested prior to starting dryout so you know they work and are accurate.

During dryout it is common to see a pattern of small (1/16-inch wide) cracks develop in a monolithic refractory lining. These cracks are an expected result of shrinkage and should not be considered a problem.

2747 Adjustment of Normal Dryout Procedure

A few situations call for the normal dryout procedure to be modified. These situations develop from the following:

- Water washing of equipment in the early stages of an FCCU shutdown
- Hydrostatic testing of refractory-lined vessels
- Hydroblasting of a tube coil or other equipment during a shutdown
- Formation of ice crystals in linings (caused by sub-freezing weather during a shutdown)

Except for the case of ice crystals, only minor modifications, if any, of the normal dryout-startup procedure are needed to compensate for the extra water absorbed into the lining. For example, if the normal startup procedure for an FCCU uses a heating rate of about 100°F/hr with a hold period when torch oil is added, no extra precautions are recommended. If the normal startup procedure is faster than that, we recommend allowing a little extra time to dry out the lining.

If ice crystals have formed in the lining (ambient temperatures below 32°F), more stringent precautions are required. Rapid heating of the lining must *not* occur until all ice has been converted to water. (See Section 7 of IRM-MS-4786.)

Also, water washing of refractory linings should not start until the hot face temperature is below 350°F. This delay is rarely a problem, because most washing is done by workers inside the unit which must be cool enough for personnel entry.

Rheniformer Lining with Shroud. No adjustment of the normal dryout schedule is needed. The recommended schedules are conservative enough that the presence of a stainless steel shroud has no practical effect on dryout.