# Technique & Application

This manual provides useful hints and application advice for using Process Temperature Control Rings (PTCR).

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## 1. What is a PTCR?

A PTCR is a ceramic device that registers the total amount of heat transferred to it. Due to the advanced techniques and materials used in its production, it provides an accurate representation of the heating process occurring in a kiln at the location of the ring. The PTCR helps to achieve a consistently high level of reliable and outstanding quality.

The actual set temperature of a kiln during a firing process, as measured by thermocouples, does not give any guarantee of the repeatability of process conditions which of course will harm the quality of the products. A thermocouple merely gives a specific spot temperature, which is not the same temperature as the one at the position of the product. The ultimate temperature of the product is determined by the total transferred heat. This is not measured by the thermocouple. Besides, the same set temperature in different kilns or kiln layouts does not guarantee the same heat treatment.

The PTCR was developed to provide a more accurate representation of firing processes, enabling precise control of the amount of heat transferred while accounting for the aforementioned factors.

The PTCR measures the total heat transferred to the ring through conduction, convection, and radiation. It considers various firing conditions that may arise in a kiln, such as heat sinks, temperature gradients, gas flow, heat transfer coefficient, time, and kiln setting. Total heat measurement ensures process repeatability and product quality.





# 2. How does a PTCR work?

The PTCR is a ceramic ring which shrinks if it is exposed to heat. The degree of shrinkage depends on the actual temperature in the kiln, the heat transfer properties of the kiln and the time it is exposed to the heat treatment. The amount of contraction, which is the reduction in the ring's diameter, can be measured using a numerical or digital micrometer.

To simplify and facilitate comparison, the total absorbed heat quantity and shrinkage are expressed as a single fictitious conversion parameter known as the 'ring temperature' (RT). It is important to note that the RT is not an actual temperature, but rather a parameter that varies with time and temperature. The RT does not indicate the maximum temperature of the firing process, nor does it correspond to the actual kiln temperature. This is because the PTCR accumulates the absorbed heat quantity over time.

The measured shrinkage is converted by means of a chart into a Ring Temperature (RT) which is an effective temperature that describes the total amount of heat absorbed by the ring and by the products. The observed ring temperature is an effective temperature for the total firing cycle, which has neither direct relation to the absolute temperature in degrees Celsius, Fahrenheit or Kelvin nor any direct relation with the set temperature. The PTCR temperature reading is further influenced by the top temperature hold time (soak time). At a constant temperature the PTCR will continue to shrink, leading to a higher RT reading. There is a maximum to the hold time, during which the ring will continue to shrink. The maximum hold time, which is typically between 0.5 and 10 hours, depends on the type of ring used and the actual temperature compared to the temperature range of the ring under consideration. The maximum hold time for ZTH rings is 2 hours, for ATH 5 hours. Using the PTCR beyond this maximum hold time may lead to erroneous results, because the PTCR ceases to shrink further on a certain moment.

# 3. Which types of PTCR are available?

There are eight different types of rings available, covering a temperature range from 560°C to 1750°C. Each ring type is colour coded and embossed with a batch number and type.

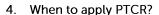
| PTCR type* | Temperature range (from – to) |
|------------|-------------------------------|
| ZTH        | 560°C - 660°C                 |
| ATH        | 600°C – 850°C                 |
| UTH        | 660°C - 900°C                 |
| ETH        | 850°C - 1100°C                |
| LTH        | 970°C - 1250°C                |
| STH        | 1130°C - 1340°C               |
| MTH        | 1340°C - 1520°C               |
| HTH        | 1450°C - 1750°C               |

\*All ring types except ZTH, ATH and UTH are alternatively available in the thinner L-variant - e.g. ETL 3.5 mm instead of 7 mm high.

Select a PTCR type whose central temperature spread is nearest to the kiln's peak firing temperature. PTCR are most sensitive to temperature changes in the central temperature range rather than at the extreme ends. If the maximum temperature is within the limit range of two ring types, the selection falls according to the holding

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time and atmosphere, since both factors also strongly influence the shrinkage of the ring.



Because of its specific properties, PTCR has a wide range of different applications for different purposes. It is a perfect means for the determination and maintenance of the exact heat treatment by the product.

## The main application purposes are:

- A) Process control and optimization
- B) Trouble shooting
- C) Yield improvement
- D) Improvement of product quality
- E) Reduction of inspection time and costs
- F) Quality assurance

## A) Process control and optimization

The most frequent use of PTCR is in optimizing and managing the firing processes to arrive at the optimal product quality and to assess the thermal performance and characteristics of a kiln.

The way of working is to make several runs with different settings of the parameter's temperature and time. These runs are characterised by the RT values as determined from the rings. Product quality analysis determines the optimal run and accompanying RT values.

In this way the found Ring Temperature is related to a specific firing process. Once the optimal setting of the process parameters is found, the PTCR is used to control the process and to recognize deviations in time and temperature. In future runs the same RT reading will guarantee the best reproducibility of the firing process.

This method is followed whenever a new product or process must be introduced, to determine differences in the thermal characteristics of new kilns, or after maintenance of kilns.

# B) Trouble shooting

This is one of the traditional applications of PTCR; malfunctioning of (parts of) the equipment. The ring is used to make a mapping of the kiln in RT in order to locate positions with different thermal behaviour.

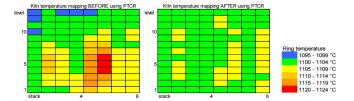
After root cause analysis proper corrective action can be taken to resolve the problem(s) after the problem is solved. PTCR are used to monitor the thermal performance of the kiln to signal the problem. In the case of monitoring the number of rings of course is smaller than during the analysis phase.

The PTCR WEB APP is the ideal supplement for the Process Temperature Control Rings PTCR. The web-based application bundles recording, documentation and evaluation simply and safely - also optimized for mobile end devices.

# C) Yield improvement

Once the mapping of the thermal characteristics is available, one has the possibility to further fine-tune the process settings and optimize SCHUPP.
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the yield. This will also lead to optimization of the energy consumption.



# D) Improvement of product quality

Because the process is controlled in a much better way, the scatter in the quality of the products is minimised. This means that the quality control of the products can be reduced as a start and may eventually be reduced further to sampling level only or even abandoned completely. Rework (if applicable) is minimized.

## E) Reduction of inspection time and costs

The improved processing will allow the reduction of the number of products that have to be inspected and may finally lead to elimination of the product testing completely. The only thing left is the measurement of the rings to monitor the process. Easy measurement can be done by using the PTCR micrometer which allows only one measurement per ring in order to get a reliable result. In time the sampling rate may be reduced to a bare minimum, reducing the inspection effort to a minimum, whilst guaranteeing an excellent product quality.

# F) Quality Assurance

Process control, as formulated in ISO 9001:2015, nowadays can only be achieved by virtue of the availability of an objective means to monitor and control firing processes. PTCR provides this objective method.

# 5. PTCR Application

# 5.1 General application advice

The general procedure for the application is as follows:

# Step 1) Placement of the rings in the kiln

Select rings with the appropriate temperature range for your application. They can be placed almost anywhere in the furnace to measure thermal performance. There is no need to measure the rings before placing them. Differences are statistically corrected and included in the conversion table. Sticking of the rings is prevented by placing a small amount of aluminum oxide powder or silica powder underneath the ring.

# Step 2) Firing process can take place

After being placed in the desired position in the kiln, the firing process begins.

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# Step 3) Measurement of the rings

After the firing cycle is complete, measure the diameter of each ring. Ensure that each ring is identifiable by its position in the kiln for mapping purposes. Each ring should only be measured once using the PTCR Micrometer, which provides reliable and accurate readings independent of the operator. In order to assure the reliability, the diameters have always to be measured between the two Ts. Rings from new batches already have a new stamp impressed with predefined measuring points (see following 2 pictures).





Step 4) Conversion of the diameters into RT

The outer ring diameter can be converted into a Ring Temperature (RT) using the conversion table delivered with every new batch of PTCR. The RT value thus obtained is an accurate representation of the total (cumulative) amount of heat transferred to the products during the temperature cycle.

# 5.2 First time usage

When applying the PTCR for the first time, it is necessary to conduct multiple trial runs with different parameter settings and compare the resulting products to the quality standard. This process helps to determine the optimal process settings and RT values. In subsequent runs, if the RT values remain the same, it can be assumed that the firing conditions were the same and the product quality is assured.

It is important to note that RT values do not necessarily correspond to absolute temperatures. A longer soak time at the same top temperature can cause the ring to shrink further, resulting in a higher RT value. However, the PTCR allows for accurate determination of differences in heat treatment both within and between certain kiln loads.

The identical measured RT values ensure an equal amount of heat transfer. This means that the firing conditions applied were exactly the same.

# 5.3 PTCR Micrometer

The PTCR Micrometer is specially developed for the measurement of the outer diameter of each ring. Because of the unique metal construction on which the Micrometer is attached, each ring has each time exactly the same position. These results in one-time-only measurements while the reliability and accuracy are assured. Other micrometers can be used to measure the diameter, but they may result in less accurate and reliable results. Therefore, we strongly recommend investing in the official PTCR micrometer.

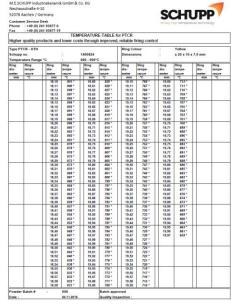




# 5.4 The temperature conversion table

The shrinkage of the diameter of the ring has to be converted into the Ring Temperature (RT). This table is updated for every batch of rings to be delivered which means that both the product and the conversion table have a unique relation.

By this re-calibration, all possible effects caused by differences in raw material(s) and deviations of green (zero hour) diameter value are compensated for. This ensures the reading of the shrinkage always to result in the same RT value and thus eliminating batch to batch differences for PTCR. This is one of the reasons why the repeatability and reproducibility of the RT readings is very high. It is important to keep in mind that mixing up of tables and PTCR batches leads to false readings of the RT value



Example of a temperature conversion table

# 5.5 Interpretation of the ring temperature

The main purpose of PTCR is to ensure the repeatability of the firing process and to ensure in this way the constant quality level of the different kiln loads. The Ring Temperature (RT) represents the total amount of heat transferred to the products and thus a constant RT value ensures firing conditions to be identical. The importance of the PTCR is to provide an accurate determination of DIFFERENCES in total heat transferred and to monitor any deviation in the processing

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conditions. The RT reading has not necessarily a direct relation to the absolute temperature. Under certain conditions RT readings may be related to the absolute temperature scales.

The best approximation without process-specific calibration is attained by heating at a rate of 120°C/hour and holding for 1 hour at the target temperature.

# 5.6 The PTCR WEB APP

The PTCR WEB APP is a web-based application. It opens many new possibilities that simplify your work with the PTCR Process Temperature Control Rings. First, the furnaces, the furnace programs and the individual firings can be stored in the application. The type of PTCR used is stored once in the furnace program. The batch used and the measurement results of the rings can now be stored in the program for each furnace run. The assignment of the batch-specific reference temperature takes place automatically and saves handling with the temperature table. Detailed information can be found here: https://schupp-ceramics.com/en



For PTCR customers of M.E. SCHUPP Industriekeramik GmbH the use of the PTCR WEB APP is free of charge!

# 5.7 Different atmospheres

The PTCR originally has been developed for use in heating processes in air. However, the ring may be applied under different atmospheric conditions as well provided some rules are considered. We advise clients to contact our customer service desk in case of using the rings for firing processes under reductive or vacuum circumstances. In this way the specific advice can be given in order to get optimal results.

Generally, it can be stated that, under vacuum conditions and reductive atmospheres like N2/H2 mixtures, the rings have to be pretreated to burn out the organic binder system of the ring completely. If this is not done properly, the organic material will not disappear as  $CO_2$ , but will decompose to carbon. This carbon, left behind in the ring, will inhibit the shrinkage of the ring, resulting in a lower RT reading than expected. Under vacuum conditions, the carbon may even precipitate on the kiln wall and contaminate the kiln.

In addition to that, under vacuum the RT reading will be lower than expected, because the heat transfer mechanism lacks the contribution of convection.

It is known that PTCR absorb carbon when used in a carbonising atmosphere. The carbon migrates through the ring and will inhibit the shrinkage significantly. It has to be determined by trial and error, to assess whether sufficient shrinkage capability is left to allow an accurate determination of the change in the outer ring diameter.

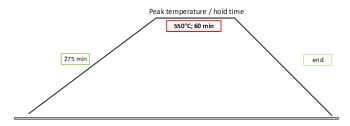


# 5.8 Pre-firing / -treatment of PTCR

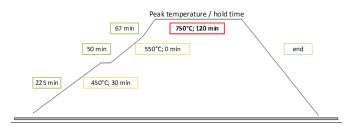
As mentioned under point 5.6 of this manual, it is absolutely necessary to pre-fire PTCR under specific requirements before using it e.g. under reducing atmospheres or vacuum. We recommend using the PTCR variants UTH, ETH, LTH and STH pre-fired whenever you want to use the PTCR outside oxidizing atmospheres. This also applies to the thinner L-version of these ring types.

PTCR ZTH, ATH, MTH and HTH are already pre-treated and do not need to be pre-treated again! Would you like to pre-fire PTCR yourself, please use the following ring specific pre-firing parameters:

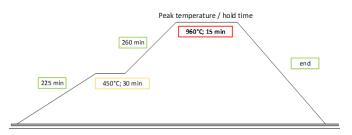
## 5.7.2 Firing curve for PTCR UTH



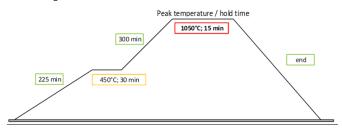
# 5.7.3 Firing curve for PTCR ETH



# 5.7.4 Firing curve for PTCR LTH



## 5.7.5 Firing curve for PTCR STH



We will gladly manage the pre-firing process for you. Please feel free to contact us!

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# 6. FAQ – Frequently Asked Questions

# 1. What is a PTCR?

PTCR is a ceramic device, which measures the total heat transferred during a firing cycle. It is not a temperature indicator in the same sense as a thermocouple, it is an energy meter. The indicated Ring Temperature is a standard that must be the same by rehearsed firing processes under the same conditions. In this way the stability of the process and quality of the products is assured.

2. What is the advantage of PTCR in comparison to thermocouples?

PTCR measures the thermal conditions at the same place where the product is located. It measures heat instead of a spot temperature. So, it measures the true important parameter for a firing process: the total amount of heat applied to the product.

3. Why is the temperature reading from the PTCR different from the set temperature?

A PTCR detects the actual amount of heat input, which depends, among other things, on the set furnace temperature, the atmosphere, and the holding time. The ring responds to the specific process conditions and shrinks as long as it is exposed to heat in and above the ring-specific temperature range. Accordingly, for example, a longer hold time results in a higher heat input, whereas the absolute temperature of the process remains unchanged. The PTCR reference temperature can therefore not be equated with the absolute furnace temperature.

4. What is the difference between PTCR and its main competitors?

The PTCR allows a numerical determination of the kiln performance and is one of the most reliable products of its kind available on the market. It assures the reproducibility of the heat treatment applied to the product and so it's quality.

5. Why is the temperature reading in R(ing) T(emperature) instead of Celsius or Fahrenheit?

Since the ceramic temperature indicator measures heat instead of temperature, the RT reading has no direct relation to the absolute temperature in Celsius or Fahrenheit. Only after taking into account both time and set temperature, a relation between RT value and the Celsius or Fahrenheit scale can be determined.

6. Can PTCR be used in every atmosphere?

In principle yes. However, in case of reducing atmospheres or vacuum conditions, PTCR UTH, ETH, LTH and STH versions of PTCR have to be pre-treated in air (see point 5.7). PTCR ZTH, ATH, MTH and HTH are already pre-treated as a standard. After this pre-treatment the PTCR will maintain its full capability without any exception. We advise to contact our customer service desk before using the PTCR in these conditions.

# 7. <u>Is the performance of the PTCR influenced by the atmospheric conditions?</u>

Yes, the atmosphere has an influence on the shrinkage of the PTCR, as atmospheric conditions affect heat conduction in the furnace. This however has no influence on the reproducibility of the reading, which will still assure the firing conditions to be within the process limits.

8. <u>Do I need to pre-treat all PTCR types before using it under reducing atmospheres or vacuum?</u>

PTCR ZTH, ATH, MTH and HTH are already pre-treated as a standard. These ring types do not need to be pre-treated again.

9. How is the performance of PTCR affected by the soak time?

The PTCR will continue to contract as long as it is exposed to heat. Effectively this means that at a constant set temperature, the RT reading will be higher, the longer the soak time is. The best approximations are obtained by applying a ramp up speed of 120 °C/hour and a top temperature hold time (soak time) of 1 hour. For a hold times of more than 1 hour please ask for the correction graph. If you work with extreme short or extreme long soak times, please contact our technical service staff. The maximum hold time for ZTH rings is 2 hours, for ATH rings 5 hours.

# 10. Do I have to adjust my kiln setting once I apply a new batch of PTCR?

No, provided the table delivered with a new batch is used, no changes to the settings are necessary. Every new batch of PTCR is factory (re)calibrated to assure the same RT reading under the same kiln conditions. The tables are created for the PTCR standard process 2°C / minute heat up to target temperature and 60 minutes hold time. If your processes differ from this, we recommend calibrating the PTCR to your specific process!

11. Is there any influence of the material of the supports used?

Yes, it is known that "reactive" support material like e.g. carbon strongly inhibits the performance of ceramic temperature indicators. The support should be of a nonreactive material, like  $ZrO_2$ ,  $Al_2O_3$ , SiN etc.

12. <u>Has the temperature ramp-up any influence on the PTCR performance?</u>

Yes, it does, because the PTCR registers the cumulative amount of heat absorbed. A long ramp-up time will result in a higher RT reading than a fast temperature ramp-up.

13. Do I need the micrometer to perform the measurements?

To ensure a correct determination of the contraction of the ring, it is of vital importance, that the ring is positioned correctly during the measurement of the ring diameter after the firing cycle is completed. The special measuring device is the best assurance of a correct "fool proof" RT reading. The PTCR Micrometer is available with USB port. With a special USB cable, you can connect the PTCR Micrometer to a computer or laptop and transfer the measuring result at

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the touch of a button. There is no special software required! We do not recommend the use of callipers, as the measuring accuracy is significantly lower compared to a micrometer.

14. Why do different batches yield different ring temperatures and how are these differences compensated for?

Batches differ, there is no question about that and there is no avoiding that. The reason for this is that the materials used to manufacture the rings differ from batch to batch. There are differences in composition, grain size distribution and so on, which affect the shrinkage of the rings made with these materials. So even though the same recipe is used with every batch, there will be small but significant differences. This will be minimized in future due to the usage of synthetically raw materials.

To compensate for these differences a calibration of each batch by firing rings from the new batch together with rings from the reference batch in their standard conditions can be done. Usually slightly different diameters are found. The diameter from the reference rings gives the actual (RT) temperature.

The diameter from the rings of the new batch is correlated to this temperature. This process is repeated 8 times, each time at a different set temperature, covering the entire temperature range of the rings. This results in 8 diameters, which correlate to 8 actual temperatures. A curve is fitted through these 8 points, which is used to make the new table. For the standard conditions the differences in shrinkage between the new batch and the reference batch now have been compensated for.

Firing under different conditions (different curve, different atmosphere, hold time, etc.) might not have the same effect on rings from different batches. When this is the case, the compensation introduced must be altered in order to achieve the exact same ring temperature with different batches of rings. This is not a bad thing. It is just a modification of the calibration to suit a specific firing process. Theoretically, the rings should be re-calibrated for each process every time a new batch is used. As a service to customers a "standard" compensation, which works very well for most processes can be provided.

Occasionally, differences between batches when fired with a specific firing process get too great or a customer wants the highest possible degree of accuracy. The customer then must calibrate the new batch of rings himself.

15. Is there any expiration date for PTCR?

No! PTCR is a ceramic material and can be stored without limitation.

16. What are the perfects storage conditions for PTCR?

PTCR should be stored dry and protected against collision - preferably in original packaging at room temperature. Temperature changes should be avoided.



# 17. What is new with ZTH?

The ZTH ring is ideal for fast firing processes; oxidizing, inert, vacuum as well as reducing atmospheres. The rings are already pre-treated as a standard.

ZTH rings are produced of 100 % synthetically and sensitive raw materials. Due to this fact the rings react very sensitively and accurate to even smallest temperature changes. Handling with care is mandatory. The rings measures exactly the peak temperature applied during thermal processes in a wide range of soaking times; 5 minutes up to 120 minutes.

## 18. What is new with PTCR ATH?

PTCR ATH are already supplied from stock pre-fired as standard. The rings can be used up to a maximum of 5 hours holding time.

19. Which ring variants are labelled with hazardous substance symbols?

The following PTCR variants are labelled: UTH, LTH, LTL, STH and STL

20. Are PTCRs considered hazardous substances? What is the meaning of the new labelling for non-pre-fired rings?

Due to the presence of hazardous substances, PTCR UTH, LTH, LTL, STH and STL are labelled with corresponding hazard pictograms in accordance with regulations. However, correct use of the rings poses no immediate risk. It is advisable to avoid dust formation through abrasion and to avoid inhaling or getting dust in the eyes. After using the rings, we recommend washing your hands and disposing PTCR properly in accordance with local regulations. Safety data sheets (SDS) are available upon request.

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