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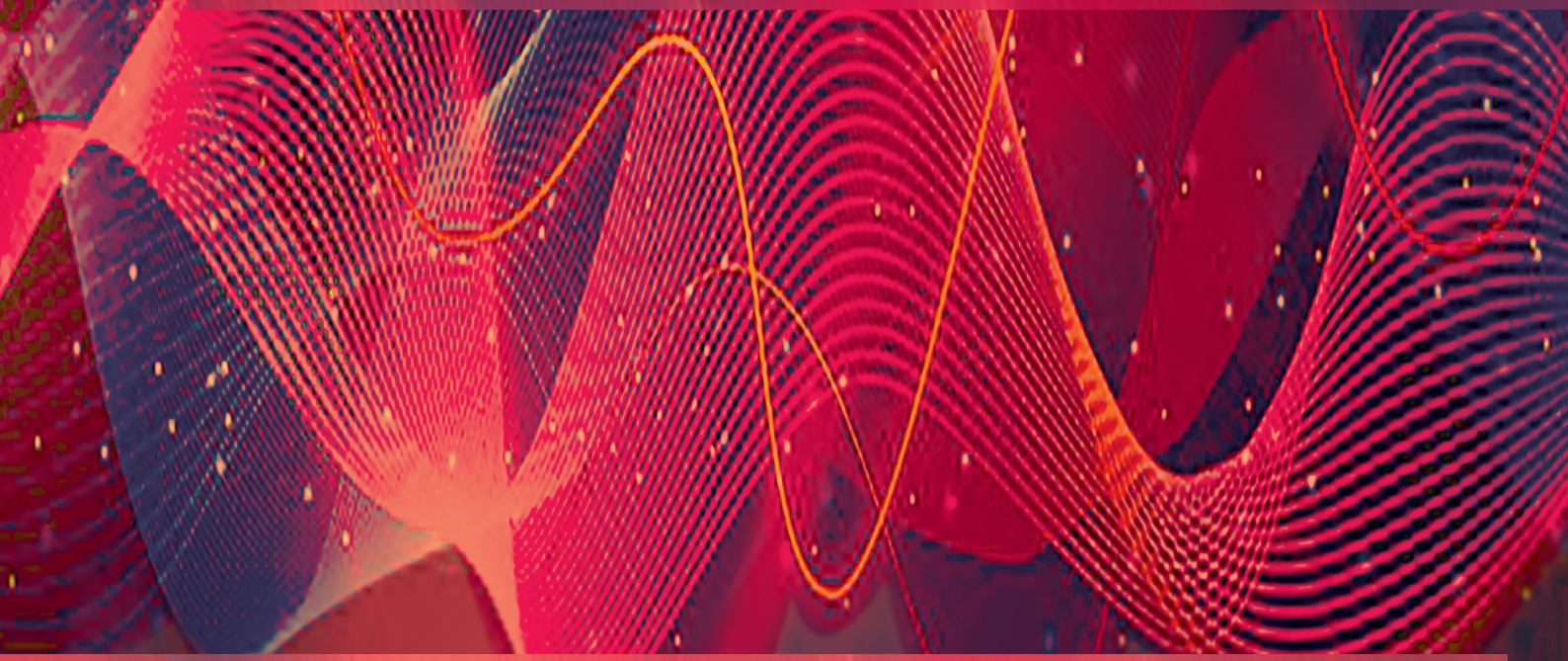
MICROWAVES IN THE SINTERING OF DENTAL ZIRCONIA



The heating of ceramic materials using electromagnetic waves is a technique used by the industry since the beginning of the 80's of the last century. Initially it was used to dry moisture in materials (basically elimination of water), and later in the heating/sintering of ceramic elements. After all this time, the technology has taken an optimal degree of maturity and more and more industrial applications are finding application on this technique.

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Focusing on the dental sector, ZrO_2 sintering is probably the most interesting utility. Right now, only a few companies in the world have mastered this technique to the point of being able to offer reliable, efficient and economical products to the market. However, due to its novelty and revolutionary concept, there are many doubts and uncertainties that assail the dental laboratory technician.

This article was born with the intention of clarifying ideas and solving doubts for a user interested in the subject, but who does not have a solid scientific basis: its character is merely informative. An in-depth analysis of microwave sintering technology would force us to tackle topics that are rooted, among others, in quantum mechanics, wave mechanics or electrochemistry. For this reason, —for the sake of clarity and ease of understanding— it has been decided to simplify.

OPERATING PRINCIPLE

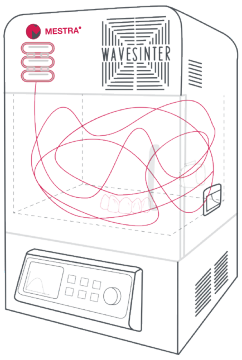
Perhaps the best way to get started in the world of microwaves is by analyzing the operation of a microwave oven alike the one we all have in our kitchen. It is known that a water molecule is made up of two hydrogen atoms and one oxygen atoms. Likewise, the hydrogen ion has a positive charge and the oxygen ion negative. Due to this asymmetry of the molecule, two differentiated areas will be generated in the space: one with a positive charge and another with a negative charge. Or what is the same: a tiny magnet.

On the other hand, we know that when a magnet is placed into an electromagnetic field, it will be oriented in the same way that the magnetic needle of a compass is oriented along the lines of the earth's electromagnetic field. But what would happen if instead of a static field like Earth's, the magnet is immersed in an electromagnetic field that oscillates in time? In that case, it would begin to vibrate at the same rate, trying at all times to maintain alignment with the field.

The food that we put in the microwave at home has a high water content. We have already seen that one way to understand the water molecule would be as a tiny magnet. When you push the start button, a device called a "magnetron" generates a high-energy oscillating electromagnetic wave. Immediately, the tiny "water magnets" begin to vibrate trying to orient themselves in the field created by the magnetron. This frenzied molecular dance produces rubbing and, consequently, heating of the food.

We must make a number of considerations:

- The frequency of the wave generated by the magnetron is extremely high. It oscillates approximately 2,400 million times per second (2,400,000,000). Molecular friction is very high.
- The electromagnetic wave penetrates into the food, reaching its interior and vibrating not only the water molecules in the outer crust, but also those found within the food.
- Because all the water molecules in the food are oscillating at the same time, heating occurs homogeneously and simultaneously throughout the mass of the food.
- As the housing of the device is metallic and closed, electromagnetic waves cannot escape from the interior (Faraday cage effect), therefore, no energy is wasted. Almost all of the energy is used to heat the food. At the end of the process, the walls of the microwave are not hot.
- There is a clear difference between heating in a classic resistance oven and in a microwave oven: in a conventional oven the radiation hits the surface of the food and from there it is transmitted to the inside of the food by conduction. For this reason, the surface of the food is toasted due to the direct incidence of radiation that is unable to penetrate inside the food. However, in the case of the microwave oven, the mechanics followed in heating prevent toasting.
- En In practice, the energizing mechanism of ceramic materials (for instance: ZrO_2) within the electromagnetic field is not the dipole type as in water (magnet). It is actually produced by shaking the free-electron/ hole pair, with oscillation space and without intermolecular crash. To energize itself, a molecule can vibrate; translate and crash into other molecules; rotate on one of its atoms; or what is more likely: a combination of all the previous actions. For reasons of clarity and simplicity in the exposition, we will continue to approach the subject following the same initial philosophy



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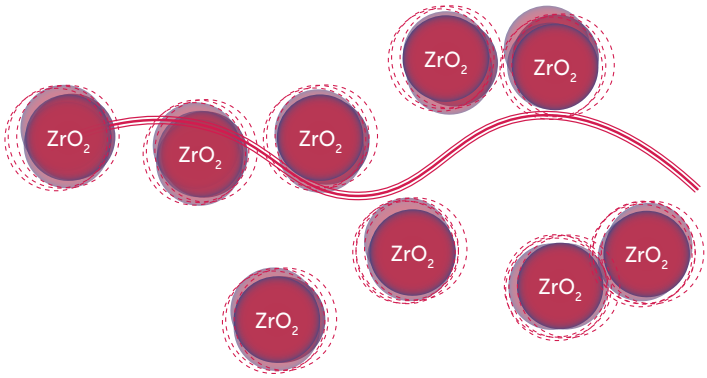
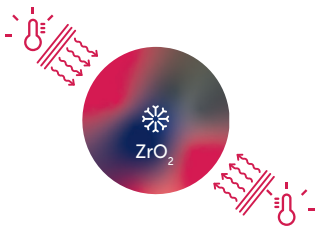
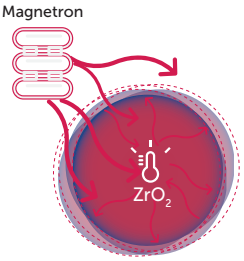
MICROWAVE MOLECULAR AGITATION



CONVENTIONAL FURNACE HEATING vs MICROWARES



FURNACE



MICROWAVES

RESISTANCES

- **In the conventional furnace, the radiation hits the surface and from there it is transmitted to the interior by conduction.**
- For this reason, **the surface is roasted** due to the direct incidence of radiation that is unable to penetrate inside.

MAGNETRON

- **Wave frecuency** > 2.4 GHz.
- **The electromagnetic wave penetrates the interior.**
- All water molecules oscillate at the same time, h**eating occurs homogeneously and simultaneously.**
- The metal casing of the device works like a Faraday cage, **the waves and heat do not go outside.**

ADVANTAGES OF MICROWAVE SINTERING

CRACKS AND DEFORMATIONS ARE AVOIDED

Let us suppose a hollow sphere that inside contains another sphere of the same material. There is initially no gap between both spheres. Starting from this condition, suppose now that we only heat the outer sphere while keeping the inner one cool. The outer sphere will begin to expand due to the effect of heat and little by little a separation will appear between the two spheres that will become larger and larger as the difference in temperatures increases.

- If we now suppose that both spheres are strongly welded, it will happen that the outer sphere will pull on the inner one and material stretching will originate. Material elasticity will try to absorb the separation between the two spheres. Obviously, when the elastic limit of the material is exceeded and the breaking stress is reached, the material will tear, producing a crack. If instead of imagining that the outer layer is the one that heats up, we now suppose that it cools, we will reach the same conclusion: the differences in expansion between the different imaginary layers that make up a material, must be absorbed by their elasticity. If not, cracks will occur both during heating and cooling.

- It's important pointing that, depending on the geometry of the piece, the homogeneity of the material and the differential temperature, the crack can appear on the outside (visible), or what is more serious: it can remain inside the piece without anyone being able to see it and cause a break when forces are applied to the material.

- It can also happen that the tensile/compressive stress caused by the differential temperature is not high enough to provoke a break (crack), but enough to produce a permanent deformation in the material called "plastic deformation". This will happen whenever the elastic limit is exceeded, without reaching the breaking stress. If this is the case, after the heating cycle the material would be warped and unusable due to the deformations that cannot be corrected.

The aforementioned is accentuated if we take into account three peculiarities of the dental zirconium:

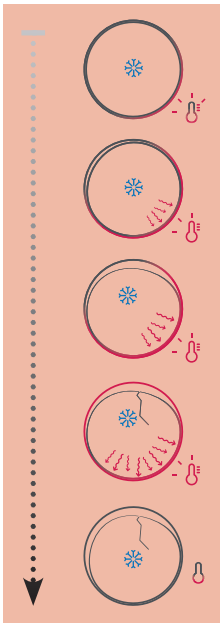
- **It is an extraordinary thermal insulator.** For this reason, the heating/cooling processes by simple heat conduction take a long time if we want to avoid temperature differences. It is difficult to conduct heat from the outside to the inside of the part, due to the high thermal reactance of the material.

- **It is a material with very limited elasticity.** Any stress produced by a difference in temperatures can cause a crack.

- **Sintered material is extremely fragile.** The ZrO_2 that we put in the furnace to sinter is not a homogeneous substance, but it is just "pressed powder". They are tiny particles of material very weakly, cohesive under pressure.

During the treatment in the furnace, these particles melt, generating a product of greater resistance. Obviously, before complete sintering is achieved, it will be a very brittle material, because the tensile/compressive strength of the "pressed powder" is very limited. Any minimal stress due to temperature difference will cause cracks.

When we heat a piece of ZrO_2 with a conventional oven, the thermal radiation produced by the resistors falls on the outer shell of the material and from there is transmitted to the inner part by conduction. The refractory character of zirconia does not aid in this heat transfer process. If we increase the radiation power to reduce the processing time, the only thing that we will achieve is to "toast" the outer crust of the piece, keeping the interior cold. Or what is worse: generating a differential temperature between the crust and the core so high, that it provokes a crack. In summary, heating/cooling times with conventional sintering furnaces must be kept long.



However, because microwaves **are able to penetrating about 30/40 mm in zirconium,** the molecular oscillation —and therefore the heating of the material— will be carried out uniformly at all points of the piece. In this way, **the risk of cracking due to the difference in temperatures between different zones is eliminated.**

SIGNIFICANT REDUCTION IN CYCLE TIMES

By employing microwave technology, processing times are also significantly reduced:

- By eliminating the need for slow and progressive heating of the material to avoid breakage and deformation, it is not necessary to extend the heating time, because there are no significant temperature differences between the inside and outside of the piece.

- The refractory character of zirconia does not have as much incidence in the cycle, because the heating originates in all the points of the material. There is therefore no need to transmit it from one area of the part (exterior) to another (interior).

- The furnace has a lower mass and also remains hot for less time than in conventional sintering. Therefore, the thermal inertia is much lower in the microwave oven. This point is especially relevant during the cooling phase: room temperature will be reached in less time than with a conventional ovenn.

- The energy transmission speed of an electromagnetic wave in matter is higher than that radiated energy. According to the laws of quantum mechanics, the photon absorption mechanisms are different in both cases and consequently also the rate of temperature change.

ENERGY SAVING

Conventional zirconia furnaces are built with resistors that radiate energy in all directions. To prevent energy losses through the walls of the furnace, thick thermal insulation systems must be used. Of all the energy emitted by the resistors, only a fraction is absorbed by the part that is heated. The main part is lost as heat in the muffle and equipment. Electric consumption is high.

However, using microwaves, the energy produced by the magnetron is concentrated almost exclusively on the material to be heated. We said that after a heating cycle in a domestic microwave, the walls are not hot. Users of our microwave zirconia oven confirm that the cost reduction on their electricity bill is significant.

SIMPLE MAINTENANCE

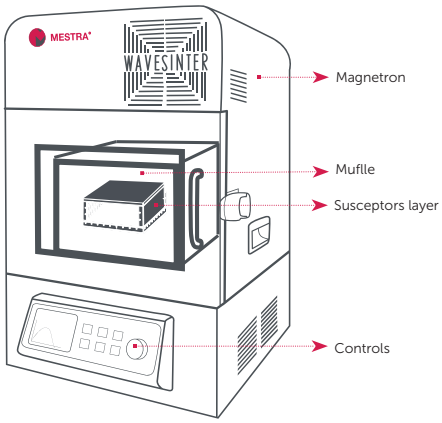
The resistances of a conventional sintered zirconium furnace have to reach temperatures that in some cases exceed 1800 ºC. These resistors are generally constructed of $MoSi_2$, an expensive material; delicate (it is very fragile and brittle); used only for very specific and limited applications; and liable to produce contamination by vapor inside the muffle.

In addition, they have a high electrical conductivity, so the use of very high currents is required (they can exceed 100 Amps), with low voltages. This characteristic requires the use of very sophisticated electronic control and power systems. To further aggravate the problem, the resistors deteriorate over time and must be replaced. Its cost is very high.

For its part, the magnetron of a microwave oven is a compact element, easy to replace in case of failure, that it cannot produce any type of contamination inside the muffle, relatively inexpensive, with a theoretical unlimited life (depending of the conditions, but it is a long life), reliable and highly tested in all types of industrial systems.

WHAT ARE SUSCEPTORS?

A recurring question frequently asked by our customers and friends is the function of the refractory susceptors placed inside of the microwave oven muffle. We will try to explain it as a curiosity. For this, we need to introduce two basic concepts: the greenhouse effect and the energy radiation mechanism.



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● Starting with the first of the concepts, we will say that a greenhouse is a glass enclosure exposed to solar action. The sun's rays pass through the glass because it is transparent at the solar emission frequency. Once inside, the radiation heats the interior, raising the temperature. This increase in temperature causes the interior of the greenhouse to in turn emit energy to the outside.

Now we must mention an important detail: solar radiation has a very high emission frequency because it has been produced by an element, the sun, which is at an enormous temperature. On the contrary, the radiation emitted when the interior of the greenhouse is heated is low energy class, because the temperature is also low. The sun's rays pass through the glass easily and heat the inside of the greenhouse, but the same glass prevents the reflected radiation from leaving. The energy enters, but does not leave, raising the temperature inside.

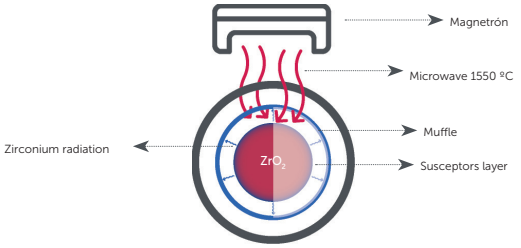
● The second of the concepts was introduced by the Austrian physicist Ludwig Boltzman, who stated that the energy emitted by a hot body was proportional to its temperature raised to the fourth power ($E = \sigma T^4$). The sun is at a temperature of several million degrees. If we raise it to the fourth power, it is easy to intuit the enormous amount of energy that radiates into space and the reason why it receives it on our planet despite being many millions of km. away. On the opposite, a body at low temperature will have a very limited energy emission capacity.

● Considering the two concepts, when heating a food in a domestic microwave the temperature does not usually exceed 100 ºC, so the radiated energy is very poor. But if we talk about dental zirconia, the sintering temperature is around 1550 ºC, about 15 or 16 times higher than a kitchen microwave. Raising this value to the fourth power, we have that the amount of energy radiated by a zirconium block at 1550 ºC will be about **50,000 times higher** than that radiated by the food that we usually heat in our domestic microwave ovens.

● Such an amount of heat could damage the internal mechanisms and systems of the oven as well as wasting energy. For this reason, our engineers chose to create a "small greenhouse". Let's not forget that sunlight, radio waves, microwaves, X-rays etc. they are all electromagnetic waves that meet the same principles and equations.

● Continuing with the simile, the interior of the muffle would be the greenhouse itself. The susceptors would fulfill the function of the crystal, which is transparent to the microwaves generated by the magnetron, but highly opaque to the radiation emitted by the heating of the zirconium. Energy can enter the furnace chamber through the susceptors, but not escape from it, resulting in energy savings.

OPERATION OF SUSCEPTORS AND MICROWAVES IN ZIRCONIA



Actually, susceptors are not 100% transparent to the energy produced by the magnetron, so a part of it is absorbed by them causing them to heat up. This heat produces two positive effects:

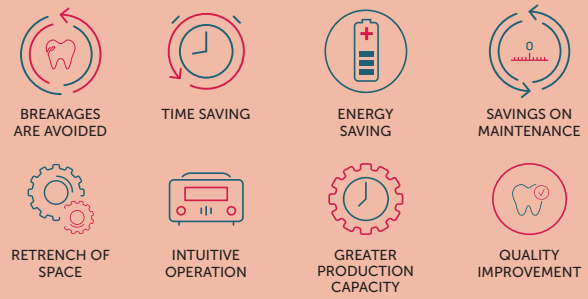
● Heats the air surrounding the sintered zirconium piece, reducing the zirconium/air temperature difference. Let's not forget that sintering is carried out in the presence of air and that air is transparent to microwaves, so it does not heat up. If there is a large temperature difference between air and the zirconium, a crack could be produced.

● In the initial moments of heating, when the free electrons in the zirconium are in their lowest energized state, the radiation of susceptors assists in the vibration of the pair free hole/electron. As the zirconium gains energy, this effect is dampened.

● Another susceptor function is to protect the electrical and mechanical systems from the radiation emitted by zirconium when it is at high temperature (about 1550 ºC). So much energy radiated at a short distance could damage the oven elements.

● As the susceptors are elements that do not suffer wear, in principle their duration is unlimited. However, the shocks and frictions produced accidentally when introducing the material to be sintered, together with the chemical contamination and the degradation of the material with the high temperature, recommend replacing these elements periodically to maintain the efficiency of the furnace. In addition, the material of the susceptors above 800 ºC can cause certain chemical reactions that deform them. This is not a problem due to the ease of replacement and the reasonable cost of the oven.

MICROWAVE SINTERING



The particular advantages of the **MESTRA** microwave sintering furnace, are not limited only to those discussed above. To them we should also add:

Compact model that occupies a small space in the laboratory. The dimensions of the equipment are practically similar to those of a conventional dental cylinder preheating oven

Simple and intuitive operation. Simple and intuitive operation. There is no learning curve. The equipment is supplied with a series of pre-recorded programs, so it is enough to press a button to start and end a cycle. The optimal result is guaranteed.

Huge production capacity. With conventional technology, a sintering cycle takes a long time, making it difficult to perform more than one cycle in a single day. As microwave technology is much faster, on the same day it is possible to launch two —and even if planned well— up to three cycles.

Significant quality improvement. Sometimes the cracks and deformations of a sintered dental material do not appear on the outside of the piece, but remain inside, being impossible to detect. However, this does not mean that there is no potential risk of breakage. It may happen that, when the prosthesis is subjected to daily use, it ends up breaking due to an internal failure that was not detected in the visual inspection after sintering. This risk is significantly reduced by using microwave heating technology.

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