THE EFFECT OF TECHNOLOGICAL PARAMETERS ON THE PROPERTIES OF ENAMEL COATING OF CHEMICAL EQUIPMENT

Emil Barta, Lampart Vegyipari Gépgyár Rt.

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The enamel coated chemical equipments take in special place in the domain of the machine manufacture for the chemical industry. The enamel coating applied on the metal surface endows the equipment with properties being workable by other technologies not at all, or in a very complicated way and expensively only. The use-value of the equipments is given by the enamel coating built-up in the course of a comparatively complex manufacturing process. The most important parameters for the users, namely chemical resistivity, thermal shock resistance, mechanical strength, resistance to abrasion etc. develop during this complex technological process. By an optimal adjustment of the multi-component system the best properties can be achieved. The least deviation from the optimal may result a deterioration of the coating properties. Coating properties depend on technological parameters, such as grain size, firing time and temperature, recooling conditions.

Owing to the complicate design (studs, double jacket, weldings, material accumulations…) at certain specific points of the equipment the properties characteristic to the enamel develop under the influence of different circumstances.

The aim of our work is the examination of the existence of these processes, further their effect on the development of the uniform coating properties.

The purpose of this paper is to look at the effect of the firing conditions on chemical resistivity and thermal shock resistance.

Examination of the firing process

Let us examine the processes taking place in the course of the firing of an autoclave. The thermovisional exposures made on the fired body show distinctly the temperature differences appearing during the cooling down of the equipment.

In **Figure 1.** the recooling curves of the mantle, studs, the bottom, can be seen. It can be observed that these preferential places recool at different speeds, in a given time moment their temperature is different too.

Similar reflected image processes take place at the warming-up during the firing. This means that certain points of the body warm-up according to different thermal curves. By the end of firing, the body takes up uniformly an identical temperature. In the course of recooling this places cool down according to different thermal curves.

Thanks to the closed design at the enamel side the temperature during the recooling can be taken as identical anywhere. This constancy is not valid viewed from the metal side.

Since the firing and recooling processes have an effect on the parameters under examination, inhomogeneities may appear within the equipment. The aim is their revelation and elimination in the interest of ensuring of a uniform coat.

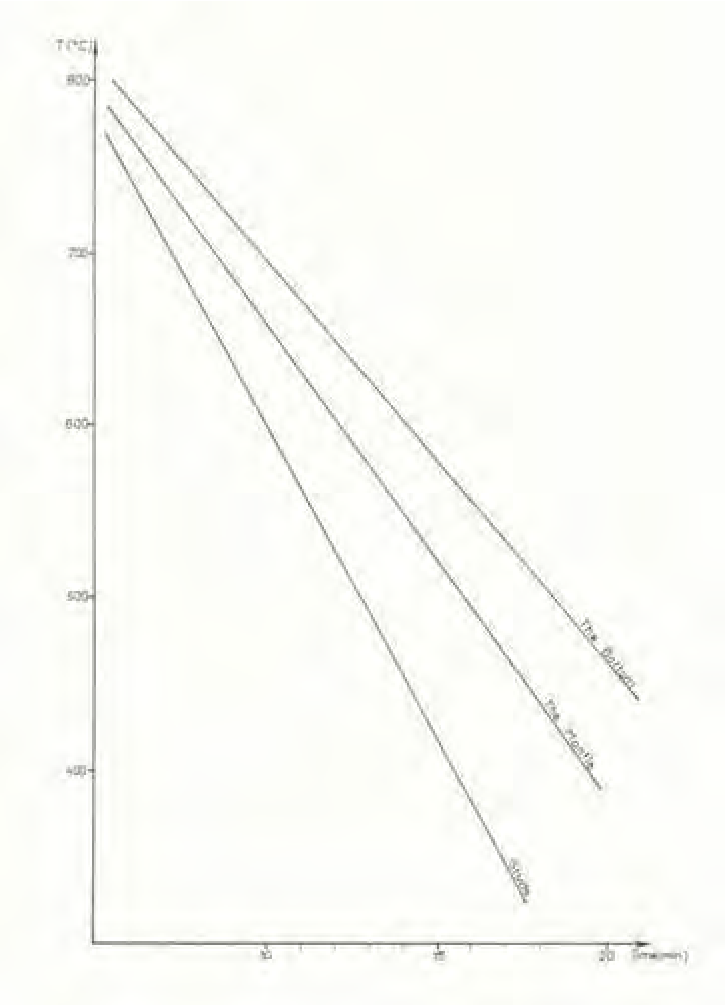

Figure 1.:
Recooling diagrams

Effect of the firing process on chemical resistivity

The chemical resistivity of the enamel depends primarily on the chemical composition, however in case of given composition the technology of firing an effect on it too.

We carried out our tests on specimens produced by the simulation of the phenomena appearing in the course of the manufacturing process, in conformity with the standards DIN 51 157, ISO 2733.

We examined the following cases:

- Effect of firing temperature
- Effect of the duration of keeping warm
- Effect of the character of firing diagram

The dependence of chemical resistivity on the firing temperature is well-known according to which the chemical resistivity improves by the increasing of the firing temperature (**Figure 2.**).

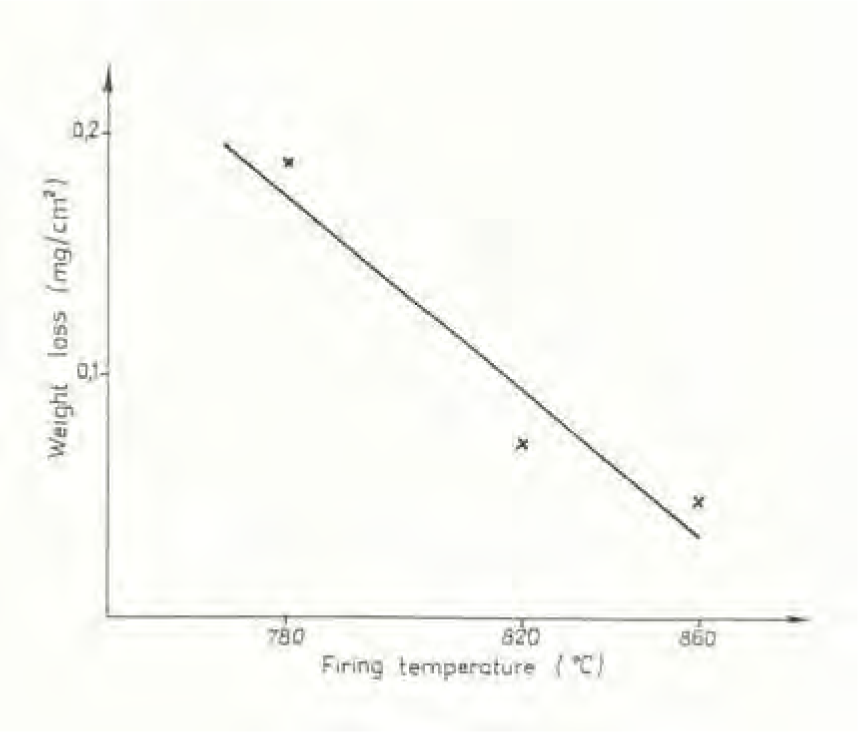

Figure 2.:
Effect of firing temperature on chemical resistivity

We fired the specimens at an identical heating-up curve for different durations (**Figure 3.**).

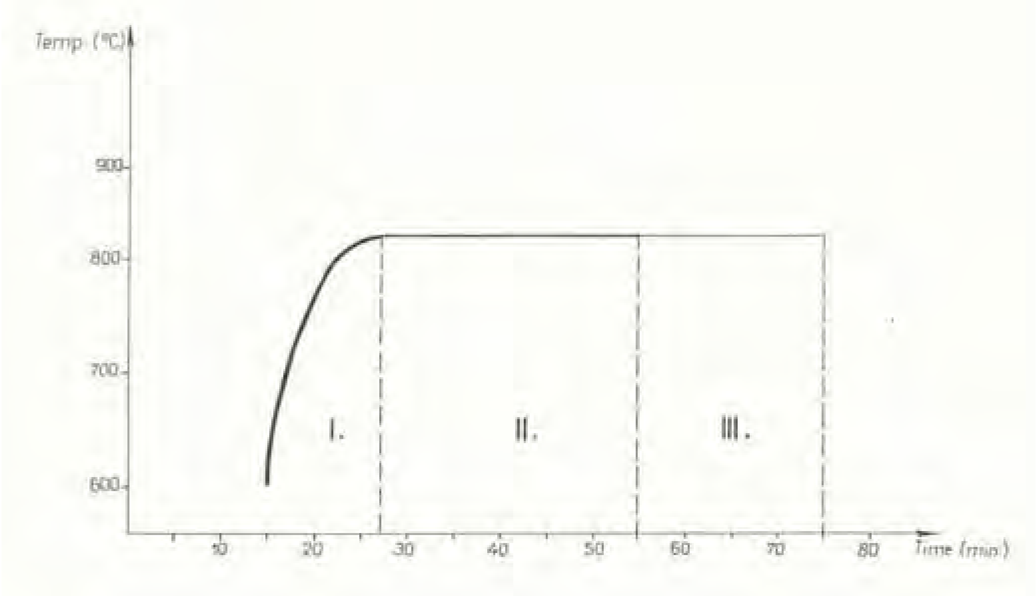

Figure 3.:
Firing diagrams of specimens of chemical resistivity

According to the results the chemical resistivity improves by the increasing of the period of keeping warm (**Figure 4.**).

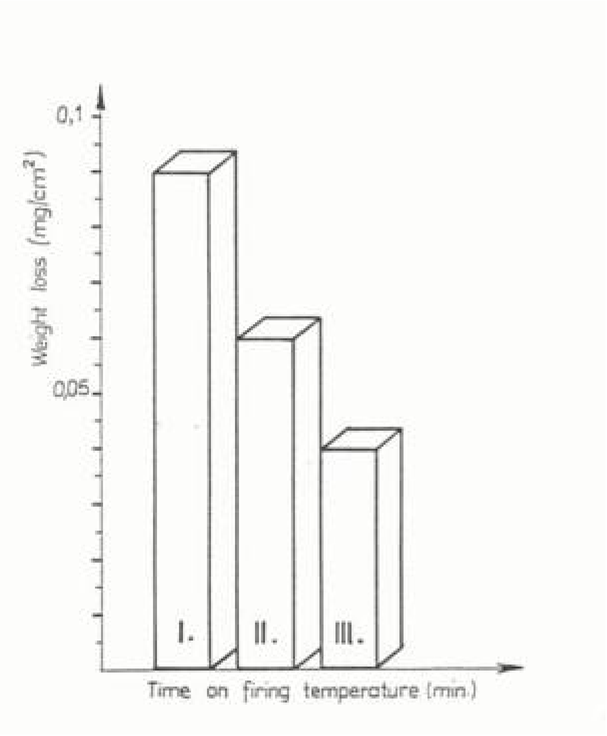

Figure 4.:
Dependence of chemical resistivity on period of keeping warm

The chemical resistivity data of specimens fired according to firing diagrams of different character showed results similar to those of the preceding cases.

In the interest of a coatdisposing of uniform properties to be ensured on the equipment, the appropriate time of keeping warm and for duration of the period of keeping warm an identical temperature at every point of the body must be ensured.

This can be obtained by putting the body in a furnace of room temperature and heating-up according to an appropriate program.

Examination of the effect of the firing process on the thermal shock resistivity

During use our enamel-coated equipments are exposed constantly to thermal effects. The service-life of our equipments is fundamentally influenced by the character and extent of the endured thermo-mechanical effects.

The resistivity of the enamel against temperature changes depends on a series of factors being determines basically by the chemical composition of the enamel (thermal expansion coefficient, tensile strength, elasticity, thermal conductivity). The effect of the minor deviation in the chemical composition appearing in the course of the manufacture of the enamel frit exerts scarcely an influence. This can be explained by the fact that we can speak of a multicomponent system, the extent of which being very small.

In addition, the resistance to thermal shock is influenced by a much more powerful factor than this.

For the purposes of the assessment of the effect of the firing-technological parameters, we prepared the specimens by simulation of the phenomena appearing in the real firing process. The measurements were carried out in conformity with the recommendation of the DIN standard 51 167.

We fired the specimens at different temperatures and different durations. The results have shown that the resistance to thermal shock passes a maximum both in function of the firing time and the firing temperature (**Figure 5.**) By applying the test results on the firing of the chemical equipments it can be said that in particular the firing at low temperature, further the firing lasting for a too long period have a harmful effect on the resistance to thermal shock.

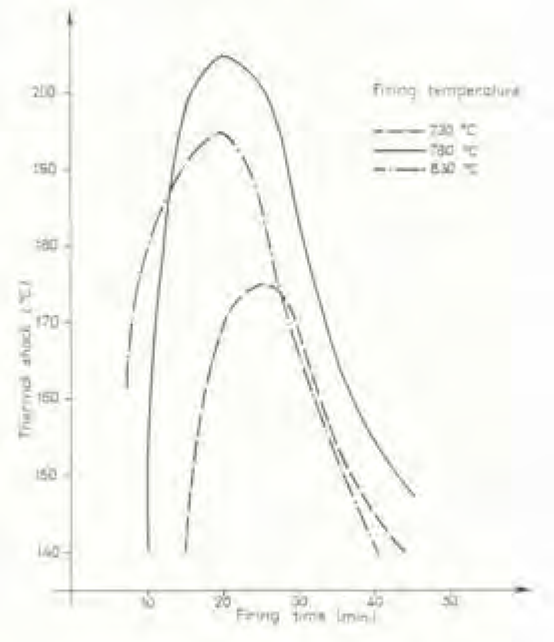

Figure 5.:
Dependence of resistance to thermal shock on firing time and firing temperature

The selection of the optimal firing time and firing temperature constitutes an indispensable condition for the developing of a coat uniform in its properties.

As mentioned already before, certain points of the body dispose of firing cycles corresponding to thermal diagrams of different character.

We fired the specimens according to the firing diagrams corresponding to **Figures 6-7.**

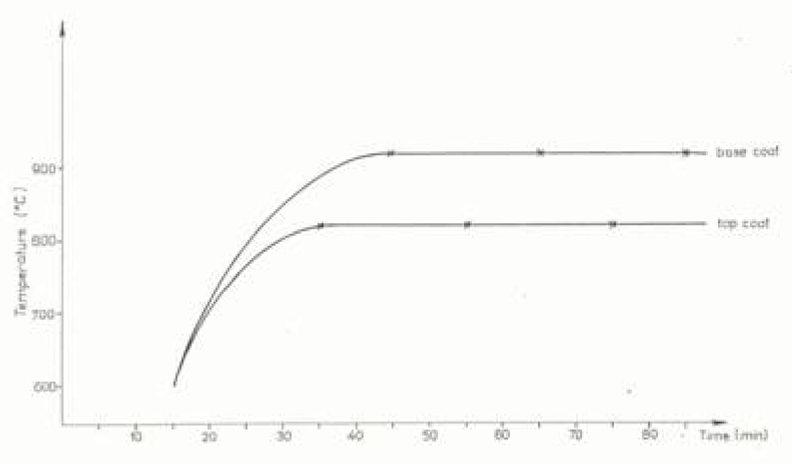

Figure 6.:
Firing diagrams of specimens of thermal shock resistance

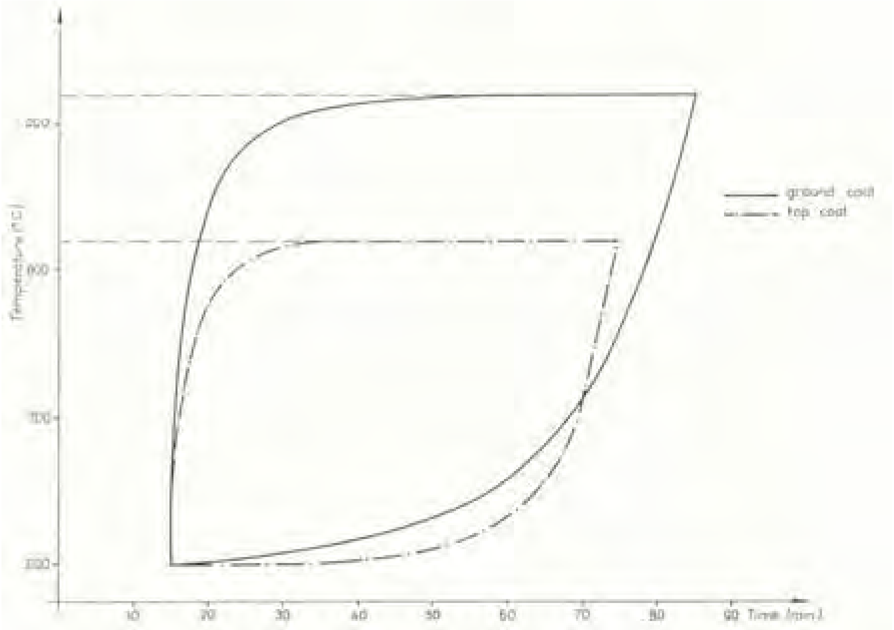

Figure 7.:
Firing diagrams of specimens of thermal shock resistance

By this simulated the effect of the extent of the period of keeping warm that is of the rate of heating-up, respectively.

The results have shown that within the given limits the effect extended on the resistance of to thermal shock does not appear. Thus homogeneity from the point of view of resistance to thermal shock is ensured on the equipments.

The resistance to thermal shock of the enamelled objects depends in a decisive way on tensions of which character prevail in the enamelling coating. The development of tensions can be traced back to different thermal expansion coefficient of the enamel and the steel.

Both from the surface of the enamel and the steel the cooling-down of the enamelled body proceeds from outside to inside. Consequently during the cooling down process the temperature proceeds in the material of the enamelled object both in space and time in a different way

to the final state. In the course of cooling down at first a superficial layer of the enamel coating solidifies. Then in the interior of the layer the enamel is still of liquid state. It follows from this that the contraction of this superficial layer is not impeded then by anything yet. Later the internal parts so to say “freeze on” this superficial layer. The contraction of them held back by the formerly solidified parts to a certain extent.

Starting out from the metal of a heat conductivity better by orders of magnitude, the conditions develop in a similar way. The meeting of the solid part getting more and more thick from the two side out, takes place somewhere in the interior of the enamel. Only from this moment on, the compressive effect of the metal extends to the full cross-section of the coating.

It follows logically from those sketched above that the state of tension in the coating is not homogenous, shear stresses are in action between the layer rows corresponding to the recooling phases. At room temperature the enamel is under compressive stress (**Figure 8**).

When starting out from the assumption that the expansive behaviour of the metal is constant, the enamel thickness and elasticity modulus are constant, the compressive stress and with it the resistance to thermal shock is a function of the congelation temperature of the enamel. This is valid if the enamel and the metal cool down together. This ideal state however occurs in practice only rarely.

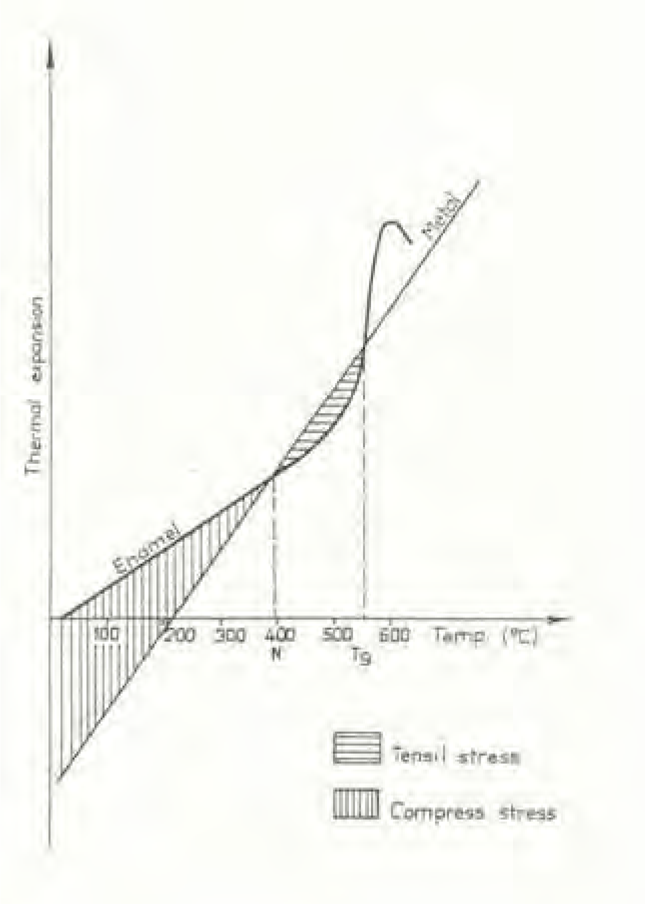

Figure 8.:
Heat expansion diagrams of enamel and metal

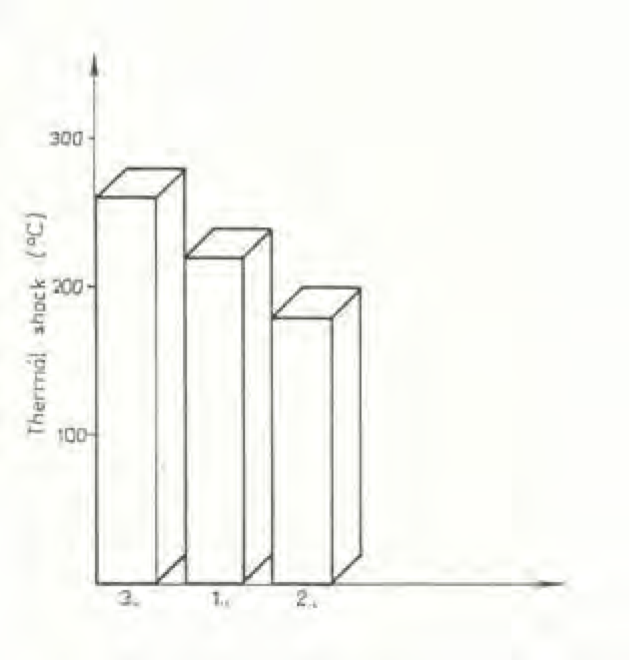

Figure 9.:
Dependence of resistance to thermal shock on recooling conditions

Distinct velocity differences appear in the resistance to thermal shock. In order to simulate the phenomenon we prepared specimens, which we cooled down at different speeds from the firing temperature.

(1) We let cool down the specimens on air, by themselves, at speed of abt. 50 °C/min
(2) We cooled down in furnace in controlled way at speed of abt. 1°C/min.
(3) We accelerated the cooling of the enamel while we delayed the cooling of the metal

The results have shown the resistance to thermal shock of the specimens of controlled cooling has weakened, while that of the specimens of accelerated cooling increased (**Figure 9.**).

Therefore the higher the metal temperature has been, at which the enamel solidified, the better has been the resistance to the thermal shock. The high resistance to thermal shock is the result of the developing of high compressive stresses.

The inhomogeneity of stress condition is further increased by speed differences of cooling down resulting from the construction. By the cooling effects of different intensity appearing on part of the enamel and the metal, the stress condition is fundamentally changed.

Some other statements still on the compressive stresses:

- The higher the compressive stress induced in the enamel layer during the manufacture will be the lower the temperature of the equipment has been
- At high operating temperatures the compressive stress in the enamel drops and the inclination to tensile stress increases
- The compressive stresses must not exceed a certain extent otherwise at convex rays a peeling-off is to be taken into account.
- In the course of the equipment an abrupt cooling –down at the enamel-side, or an abrupt warming-up at the metal-side result in a decrease of compressive stresses, while an abrupt cooling-down at the metal-side, or an abrupt heating-up at the enamel-side result in an increase of the compressive stress.

The areas endangered as a consequence of the inhomogeneity of the compressive stresses are the outer and inner radii of the studs, the parts enamelled from outside. At these places the process **(3)** takes places.

By suitable selected cooling technology the inhomogenities arising from the construction can be reduced to a minimum level, thus the reliability of the equipment regarding resistance to thermal shock can be increased. This can be accomplished by a regulated cooling of the fired body in a cooling chamber.

Summary

It can be established that while the chemical resistivity is mainly a function of the firing process, the resistance to thermal shock depends in a decisive way on the cooling process.

By selecting optimally the firing time and the firing temperature, the best values of the two properties can be achieved.

By applying an appropriate cooling technology the resistance to thermal shock thus obtained can be preserved uniformly regarding the equipment.

It can be established that the spontaneous processes developing during the manufacture may change the properties characteristic to the coat to a considerable extent, at about 20%.

Therefore it is worth to keep an eye on them, even if it is to the expense of productivity, in the interest of the improvement of the quality.