VITREOUS ENAMEL COATED
BOLTED STEEL TANKS / SILOS:
PAST & FUTURE

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Vitreous Enamel Coated Bolted Steel Tanks / Silos: Past & Future

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1. Abstract
Vitreous enamelled coated bolted steel tanks / silos are on the market for around 50 years. They are used for the storage of a high variety of liquids, slurries and dry bulk products in municipal and industrial areas. Thanks to the very good chemical resistance of the vitreous enamels and the flexible and quick construction, not obtained by other silo technologies, there is a growing interest for enamelled tanks / silos. The complete production process of the panels is reviewed, including steel requirements, enamel properties and final product quality. The development of a new family of enamels for the fish scale free enamelling of non enamellable construction steel is discussed.

2. Introduction
During the last decade, the use of vitreous enamel for industrial applications has grown a lot and this application area became the second largest enamel consuming market after the household appliances.

Industrial applications of vitreous enamel are hot water tanks, silos/tanks, heat exchangers and chemical vessels. For these products, the vitreous enamelled steel is chosen from an engineering point of view because of the combination of following excellent technical properties:
- Inorganic chemical resistance over a wide pH-range
- Excellent organic chemical resistance.
- Temperature resistance till high temperatures
- Very smooth, non reactive and non sticky surface
- Scratch and abrasion resistance
- Physiological safe
- No bacteriological growth
- Strength of the enamelled steel

Different from hot water tanks, heat exchangers and chemical vessels, the surface of the silos/tanks is visible during use. For this reason besides of the technical properties, which are the most important, there also is the esthetical aspect of the colour.

3. Bolted steel tanks /silos
Bolted steel tanks/silos are made in two steps: the vitreous enamelled panels are shaped, enamelled and carefully packed in the enamel work shop and the tank/silo itself is erected in situ. In this way, the silo/tank is very fast constructed. This is a big advantage against concrete silos/tanks. Another practical advantage is the possibility to remodel, extend and dismantle a bolted silo/tank.

Besides of the wall panels, it is also possible to enamel floor panels and roof panels. In this way, the complete silo/tank is made out of the same material.

Bolted silos/tanks can be created with diameters till 100m and heights till 50m; average sizes are 6 m diameter and 6 m height. The erection of the silos can be done with an erection platform or with
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jacks (i.e. the silo is constructed ring by ring on the ground and is jacked up every time a ring is finished - especially used for roofed tanks.

The edges of the panels have to be carefully enamelled in order not to have enamel defects. For this reason the edges of thick gage sheet are rounded. Sometimes, sealants are used on the edges to overcome corrosion. The sealant has to be resistant against the used medium in the silo!

The used bolts are normally made out of stainless steel or galvanised steel, depending on the used medium and the needed strength.

Besides of vitreous enamelled steel panels, bolted silos/tanks are also made out of organic coated steel panels, galvanised steel panels, aluminium panels, stainless steel panels. But non of these alternatives gives the same excellent combination of properties as vitreous enamelled steel panels, and for this reason the vitreous enamelled panels are used for a wide range of silo/tank applications: from the dry storage of animal feed, over the storage of potable water till the use for digesters and storage of aggressive industrial effluents.

For every silo/tank, an engineering study has to be made taking into account the specifications of the medium and its temperature, the environmental conditions (wind, seismic, snow, ice, temperature range), the usage and planned dimensions of the tank, location of openings, attached equipments, etc. Out of this input, the required steel thickness/properties and the required enamel quality for the different panels are determined.

3.1 Required enamel quality

There exist a lot of standards that are dealing with the required properties of the enamel coating of bolted steel silos/tanks. The following ones being the most important:

The ISO 4528: vitreous and porcelain enamel finishes – selection of test methods for vitreous and porcelain enamelled areas of articles, mentions the properties (and corresponding standards) that are important for both silos and tanks, but without giving minimum requirements for those properties.

The EEA (European Enamel Authority) Quality Requirements specifies in §7.20 the minimum requirements for enamelled industrial tanks and in §7.24 - 25 the minimum requirements for enamelled silos for animal feed and for dung.

The new European standard EN15282 – 2007: vitreous and porcelain enamels – design of bolted steel tanks for the storage or treatment of water or municipal or industrial effluents and sludges, specifies all the requirements for the design of bolted cylindrical steel tanks and the minimum requirements for the enamel coating for the different liquid storage tank applications.

In the above mentioned standards, the following quality requirements for the vitreous enamel coating are specified: the resistance to chemical corrosion by different acid and alkaline solutions, the thermal shock resistance, the impact resistance, the abrasion resistance, the scratch hardness, the adherence, the enamel layer thickness, the defects in the enamel layer and the colour. Which properties that are needed and which minimum requirements of these properties that are needed strongly depend on the silo/tank application: table 1 gives the requirements for some typical applications.

As you can see from table 1, the most severe requirements are asked for the panels for the roof and the top ring. This is the most critical part of a tank: a better chemical resistance is needed because the vapours, gasses that are formed out of the liquids are present in this top area and are more
aggressive than the liquids themselves. And a better thermal shock resistance and impact resistance is needed to withstand rain, ice and snow fall.

The enamel layer of the floor plates has to fulfil the same requirements as the panels for the cylinder.

Depending on the required properties, different enamel coatings are used, ranging from a direct on enamel coating with good chemical resistance towards a 3 coat system with a top coat that has a perfect chemical resistance in the range pH 1-14. The total layer thickness varies correspondingly with the number of coatings. One has to avoid excessive layer thickness at the borders and around the bolt holes, in order to avoid cracks and chipping off during the construction and the bolting of the panels. The enamel is mostly applied by wet spraying or dry electrostatic powdering. The PEMCO enamel assortment contains all enamel qualities needed for silo/tank enamelling, both for wet and powder application, fulfilling the properties asked by the given standards and beyond.

### Table 1 Requirements of the vitreous enamel coating on silos

<table>
<thead>
<tr>
<th>Test</th>
<th>Test method</th>
<th>EEA, §7.24</th>
<th>EEA, §7.20</th>
<th>EN15282</th>
<th>EN15282</th>
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<tbody>
<tr>
<td>Animal feed</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Industrial tank</td>
<td></td>
<td></td>
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<tr>
<td>Potable water</td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Thermolytic/pasteurization digester</td>
<td></td>
<td></td>
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<td></td>
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<tr>
<td>Roof top ring</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Cylinder</td>
<td></td>
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<table>
<thead>
<tr>
<th>Chemical requirements</th>
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<tbody>
<tr>
<td>Citric acid Room temp</td>
<td>EN14483-1.9</td>
<td>A</td>
<td>A</td>
<td>A+</td>
<td>AA</td>
</tr>
<tr>
<td>Sulphuric acid Room temp</td>
<td>EN14483-1.10</td>
<td>-</td>
<td>A</td>
<td>AA</td>
<td>A+</td>
</tr>
<tr>
<td>Hydrochloric acid Room temp</td>
<td>EN14483-1.11, 15 min</td>
<td>-</td>
<td>A</td>
<td>AA</td>
<td>A+</td>
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<tr>
<td>Citric acid Boiling – 2.5h</td>
<td>EN14483-2.10</td>
<td>10 g/m²</td>
<td>5 g/m²</td>
<td>3 g/m²</td>
<td>0.75 g/m²</td>
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<td>Hydrochloric acid Vapour – 7d</td>
<td>EN14483-2.12</td>
<td></td>
<td></td>
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<td>7 g/m²</td>
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<td>Water Boiling – 48h</td>
<td>EN14483-2.13</td>
<td>10 g/m²</td>
<td>10 g/m²</td>
<td>5 g/m²</td>
<td>2.5 g/m²</td>
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<tr>
<td>Water Vapour – 48h</td>
<td>EN14483-2.13</td>
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<td></td>
<td></td>
<td>5 g/m²</td>
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<tr>
<td>A6-detergent sol. 95°C – 24h</td>
<td>EN14483-3.9</td>
<td>5 g/m²</td>
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<td>2.5 g/m²</td>
<td>5 g/m²</td>
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<td>Sodium hydroxide 80°C</td>
<td>EN14483-4.9</td>
<td>3.5 g/m² (5h)</td>
<td>3 g/m² (5h)</td>
<td>7 g/m² (24h)</td>
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<table>
<thead>
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<th>Physical requirements</th>
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<tbody>
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<td>Thermal shock</td>
<td>ISO 2747</td>
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<td>Impact resistance</td>
<td>ISO 4532</td>
<td>20N</td>
<td>20N</td>
<td>20N</td>
<td>40N</td>
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<td>Abrasion resistance</td>
<td>ISO6370-2</td>
<td></td>
<td></td>
<td>45g/m²</td>
<td>45g/m²</td>
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<tr>
<td>Mohs scratch hardness</td>
<td>EN101</td>
<td></td>
<td></td>
<td>Mohs 5</td>
<td>Mohs 5</td>
</tr>
</tbody>
</table>
### 3.2 Required steel quality

For the production of vitreous enamelled panels for tanks / silos, one of the biggest problems is to get enamellable construction steel with the needed high mechanical properties.

#### 3.2.1 Enamellability

Construction steel sheet is normally produced by hot rolling and does not have any fish scale resistance. For the production of hot water tanks, that are only enamelled one side, this is not causing problems because the hydrogen release can take place at the outside surface of the steel sheet of the hot water tank, so that no fish scales occur in the enamel layer at the inside. Bolted tanks and silos panels are both side enamelled and so, there is a need for both side enamellable high strength hot rolled steel sheet. This is very difficult to obtain because of the fact that no micro-voids can be created during hot rolling. It are especially those micro-voids that are created during the cold rolling, that suppress the fishscale formation of the widely used low carbon enamellable cold rolled steel qualities. For the hot rolled steel, other metallurgical possibilities have to be used. During the last decades, a lot of steel suppliers have performed research for such steel qualities [1,2,3]; almost always, the metallurgists are using titanium additions and are controlling carefully the ratio of titanium, nitrogen, sulphur and carbon. The purpose is to have less austenite (which can contain much more hydrogen) during the enamel firing and to create preferable Ti-precipitates during the hot rolling process. Although that the research results are very promising, it is very difficult to produce these qualities with homogeneous properties and so, only very few steel suppliers have these steel qualities in their assortment. And the price of these difficult producible steel sheet is correspondingly high.

Some manufacturers of vitreous enamelled steel panels are using such Ti-added hot rolled construction steel sheet successfully, the others are buying non-enamellable construction steel and are using special pre-treatment or enamel slip to avoid fish scaling. (see lower)
3.2.2 Mechanical properties
Besides of the fish scaling problematic, one should also keep in mind that the mechanical properties of the bought steel change during the firing of the enamel layer(s). The firing, that can be compared with an annealing of the steel, diminishes the strength of the steel panel. This can be very drastically, as reported in [1]: an initial yield stress of 771 MPa was reduced during enamel firing to 450 MPa. And it are these strength properties after firing that need to fulfil the calculated minimum mechanical requirements of the panels! For this reason, one should best order a steel quality with guaranteed mechanical properties after annealing.

The minimum thickness of a tank shell shall be 1.5 mm after EN15282. Most tanks, silos are constructed with sheet steel of different thicknesses: the higher in the tank, the lower is the load and the hydrostatic pressure and the thinner can be the steel sheet. So the 1.5 mm is the minimum thickness of panels for the top of the wall, the steel sheet at the bottom can be > 10mm depending on the height and the load of the construction. It is also a common practice to use steel qualities with higher mechanical properties for the bottom panels, in order to attain the needed strength with lower thicknesses. Successfully used are the steel qualities DD11, S235, S355, S420, S460 after EN10111 and EN10149-1.

For the firing of the enamel layer on the panels, the parameters have to be set in correlation with the steel thickness: the thicker the panels, the longer the firing time.

3.2.3 Pre-treatment
The steel panels need to be pre-treated before enamelling. The black iron oxide formed during the hot rolling has to be removed. This can take place in the steel plant by pickling, or can be done in the enamel shop, where both pickling and shot blasting can be used. The advantage of shot blasting is that there is a better contact area between steel and enamel, resulting in a better reaction during firing.

If the pickling takes place in the steel shop, the sheets are normally corrosion protected. This oil film as well as the lubricants used during the panel fabrication have to be removed before enamelling in order to avoid enamel defects.

A new method of brushing the steel sheet before enamelling is practiced successfully by GLS Tanks [4]: the steel sheets are bought pickled but not oiled, and the brushing removes the little amount of rust that has been formed and also activates the surface.

3.3 Existing processes for fish scale free enamelling
In order to avoid fish scaling on “non enamellable” steel qualities (with insufficient hydrogen traps for both sides enamelling without fish scaling), the following methods are reported in the literature:

- Protonic conductors mixed in the enamel coating are described in the patent FR2 784 696 [5]. The homogeneous dispersion in the enamel layer of products having a protonic conductivity higher than $10^{-8}$ S.cm$^{-1}$ at 300°C is reducing the sensitivity to fish scaling defects. These protonic conductors can be $\beta$, $\beta'$, $\beta''$ alumina or $\beta$, $\beta''$ gallium oxide. The product is added to the enamel slip at 2 to 25 weight-% and is increasing the hydrogen permeability from the glass transition temperature till 300°C. Disadvantage of this method is that aluminium oxide is diminishing the chemical resistance and adherence of the enamel coating and Gallium oxide is much to expensive, therefore the use of this technology is limited and not used for bolted tanks/silos.
- Crystalline nickel oxide mixed in the enamel coating is described in the patents US2 940 865[6] and US6 177 201 [7]. Crystalline NiO is added to the slip in such amounts that it forms 2 to 10 weight-% of the total solid in the ground coat. This enamel slip is applied directly on the steel in a single coat or in multi layers. The disadvantage of this method is the fact that crystalline NiO (CAS No 1313-99-1) is carcinogenic (Carc. Cat.1; Danger Symbol T; R43-49-53 after the European directive 1967/548/EG An. I) [8] and the storage of NiO is only possible under restricted conditions (SEVESO II-directive 96/82/EG).

- Applying an alkaline phosphate slip, rich in nickel oxide, to the steel surface as a pre-treatment [9]. Here also the use of carcinogenic NiO is restricting the use of this method.

One of the big strengths of PEMCO is that we are offering the most complete range of vitreous enamels for all possible applications and that we have specialists with long experience for all of these product ranges. For decades, PEMCO is bringing new and innovative enamel products on the market in order to help our customers solving their problems. The both side enamelling of hot rolled construction steel being an issue over the last years, we started a research project. Out of this project, a new family of enamel frits was developed with which it is possible to enamel these non-enamellable steel without fish scales.

4. Development of fish scale free enamel
In order to evaluate the susceptibility of new enamel systems to hydrogen defects, a specific fish scale test was developed combining two existing test methods.

4.1 Test method
Miller [10] developed a test based on work previous done by Deringer [11]. Both authors used a cathodic pickling setup as described in figure 1.

![Fig.1 Cathodic pickling apparatus](image)

Atomic hydrogen forms on the bare steel side of an enamelled plate. It diffuses into the steel and collects as molecular hydrogen at the glass-steel interface. The time delay between beginning of hydrogen diffusion and the first fish scale defect provides information about the steel and steel – enamel interface.
A similar apparatus was again used by Wilczynski and Wallace [12] to modify enamel systems composition and qualify enamel slip compositions with better fish scale resistance.

The European standard EN10209 uses the Ströhlein apparatus to test the hydrogen diffusion and so the enamelability of sheet steel. In this test, the hydrogen is electrolytically generated on one side of a degreased plate. The time between the start of electrolysis and the determination of hydrogen that passed to the other side of the plate is defined as the hydrogen permeation time. A long permeation time indicates a good resistance to fish scaling.

The hydrogen permeation $TH$ in min/mm² is calculated as follows:

$$TH = \frac{15t_o}{d^2}$$

$t_o$: permeation time in minutes

$d$: plate thickness in mm

When $TH$ is greater than 100 the steel lot will not show fish scale defects when enamelled on both sides.

Our new fish scale lab test combines the idea of Miller with the apparatus and calculation method of EN10209. The apparatus is described in figure #2.

The electrolysis container is temperature regulated at 25°C. Hydrogen is generated electrolytically on the bare side of an enamelled steel plate. Electrolyte is made up of 6% by volume of H₂SO₄ with 0.25 g/l of HgCl₂ and 0.5 g/l of As₂O₃. The solution shall be freshly prepared for each test. Time for fish scale defects to appear on the opposite side is recorded from electrolysis start. (Visual inspection).

A new parameter, TFS, is introduced, which is the Time for first Fish Scale to appear (in min/mm²) is calculated as follows:

$$TFS = \frac{15t_{fs}}{d^2}$$
$t_{fs}$: time for first fish scale defect in minutes

d: plate thickness in mm

In this investigation, the $t_{fs}$ values are always the average of 4 measurements. Deviation is around 15-25%. Using this new fish scale test and TFS value, various tests were done.

### 4.2 Test parameters

A standard wet hot water tank formulation was used in order to evaluate fish scale sensitivity.

<table>
<thead>
<tr>
<th>Component</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Frit</td>
<td>100</td>
</tr>
<tr>
<td>Silica</td>
<td>40</td>
</tr>
<tr>
<td>Clay</td>
<td>6</td>
</tr>
<tr>
<td>Borax</td>
<td>0,15</td>
</tr>
<tr>
<td>Boric acid</td>
<td>0,15</td>
</tr>
<tr>
<td>Sodium nitrite</td>
<td>0,15</td>
</tr>
<tr>
<td>Water</td>
<td>50</td>
</tr>
</tbody>
</table>

Frits, additives and water were ground in ball mills to milling fineness 6 on Bayer sieve 3600.

Slip was sprayed on S235J (EN10025) hot rolled steel with various pre treatments, dried and fired in a box furnace during 8 minutes at various temperatures.

The S235J hot rolled steel used for this study is having a TH value lower than 25 min/mm².

### 4.3 Influence of pre-treatment

Degreased only, grit blasting and sulphuric acid pickling are three common pre-treatments in enamelling industry. These surface pre-treatments were compared using the TFS values.

![Fig. 3 Influence of steel pre treatment on TFS values](image-url)
TFS values are low at low firing temperatures and are increasing with higher firing temperatures except for pickling. This behaviour seems to be correlated with enamel to steel bond. Bond noted according to EN10209 is reported in following graph (picture #4).

At low firing temperatures 780°C and 800°C when bond is below class 3, for all pre-treatments the TFS values are low. When firing temperature is sufficient and bond is good TFS value are higher. It means that until bond has not developed between steel and enamel, the scale defects are appearing rapidly. Blasting is showing higher TFS values than degreasing or pickling. It appears that grit blasting is offering a better protection against fish scaling compared to degreased only or sulphuric acid pre-treatment.

4.4 Influence of bubble structure

Bubble structure and its effect on fish scale resistance have already been investigated by G. E. Miller [10], C. G. Bergeron [13], S. Ali, A. Hardwick and X. Yang [14]. Authors are reporting the positive benefits of developing bubbles at enamel steel interface.

These positive effects have been reproduced and observed. A non oxide mill addition which is increasing the bubble structure during firing was added to the standard milling formula. All slips were sprayed on degreased only S235J hot rolled steel, dried and fired during 8 minutes at various temperatures.
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Fig. 5 Evolution of TFS value with increasing bubble structure

Bubble structure after firing at 840°C for 8 minutes are compared on the following cross sections.

Picture #1: 0% additive. Picture #2: 0.02% additive. Picture #3: 0.05% additive.

4.5 Influence of enamel stress
Enamel and steel have different coefficient of thermal expansion (CTE). Enamel is designed in order to have a CTE lower than steel so that after cooling at room temperature the enamel surface is under compression. Depending on steel CTE, enamel CTE and softening temperature, the amount of compression of the fired enamel layer will be different at room temperature. One way to measure this amount of compression is the Klotz apparatus (DIN51175). The Fd value is providing an indication of enamel compression at room temperature. See figure #6.
The standard enamel formula used is having an Fd value of 1.52. By replacing equal amounts of silica by zircon silicate in the standard milling formula, the stress in the enamel layer was increased. TFS values of these higher stress enamels were compared at 840°C 8 minute firing using two pre-treatment conditions.

When stress is gradually increased from Fd = 1.53 up to Fd = 2.52, the TFS value is reduced. This means that fish scale defects are more prone to appear when enamel layer is under a higher stress. When hydrogen pressure is building up at enamel steel interface, if enamel is already under high compression, the break down of the enamel will happen faster. The degradation appears with both degreasing and blasting steel pre-treatment.
4.6 Influence of enamel frit composition

The enamel frit used in the standard milling formula has been modified in order to evaluate the influence of the different metal oxide. The total amount of metal oxide was kept equal. TFS values were recorded for each modified frit using the following formula.

<table>
<thead>
<tr>
<th>Modified frit</th>
<th>100</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silica</td>
<td>40</td>
</tr>
<tr>
<td>Clay</td>
<td>6</td>
</tr>
<tr>
<td>Borax</td>
<td>0,15</td>
</tr>
<tr>
<td>Boric acid</td>
<td>0,15</td>
</tr>
<tr>
<td>Sodium nitrite</td>
<td>0,15</td>
</tr>
<tr>
<td>Water</td>
<td>50</td>
</tr>
</tbody>
</table>

Frits, additives and water were ground in ball mills to milling fineness 6 on Bayer sieve 3600.

Slip was sprayed on degreased only S235 hot rolled steel, dried and fired in box ovens at 840°C 8 minutes.

Depending on the nature of the metal oxide melted in the frits the TFS value can reach values as high as 150. TH values higher than 100 are recommended to enamel steel on both sides. The steel used for this study is having a TH value lower than 20.

Enamel steel systems having a TFS value higher than 150 have been used to enamel hot rolled steel on both sides. These enamel steel systems are not showing any fish scales.
4.7 Development of fish scale resistant enamel coatings
Based on the above results a new family of frits have been developed.

It is not needed to use 100% of these frits in the milling formula. Depending on the chemical composition of each frit only a minimum amount of these specific frits is necessary to reach high TFS value when enamelling hot rolled steel having TH value lower than 100.

The frit system developed enables also a two coats one firing technique. The base coat and top coat have following formula:

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
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<tbody>
<tr>
<td>New frit</td>
<td>25 to 50</td>
<td>TS2160</td>
</tr>
<tr>
<td>Ground coat</td>
<td>50 to 75</td>
<td>Silica</td>
</tr>
<tr>
<td>Silica</td>
<td>0 to 10</td>
<td>Clay</td>
</tr>
<tr>
<td>Suspending agent</td>
<td>2 to 8</td>
<td>Sodium aluminate</td>
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<tr>
<td>Suspension salts</td>
<td>0,2 to 1</td>
<td>Potassium carb.</td>
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<tr>
<td></td>
<td></td>
<td>Sodium nitrite</td>
</tr>
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</table>

This 2C/1F system can be applied on degreased only steel. It is preferably applied on grit blasted steel as blasting is beneficial to fish scale resistance.

As shown on figure #9 TFS value as high as 150 can be achieved with 20% of frit A, 40% of frit B or 50% of frit C.

5. Conclusion
A new family of enamel frits was developed with which it is possible to enamel non-enamellable steel without fish scales.
This new family of frits enables to enamel steel qualities that have TH values lower than 100. In the past enamelling such steel qualities on both sides was only possible by using specific mill addition in the slip or specific steel qualities.

This line of PEMCO products has been successfully tested.

References

[6] US2 940 865; Method of applying a glass coating to steel by using an intermediate layer of nickelous oxide
[7] US6 177 201; Porcelain enamel coating for high-carbon steel
[8] Ceramic Decorating Materials; ANFFECC/CERAMICOLOR/EPSOM/VdMI; 1998; p.45

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