Proceedings of the
48th Porcelain Enamel Institute
Technical Forum

Larry L. Steele
Conference Director

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Technical Forum

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A Message from the Technical Forum Committee Chairman

As Chairman of this year's Committee, I am pleased to present to you the Proceedings of the 48th Porcelain Enamel Institute Technical Forum. Again this year, just about every aspect of the processing and application of porcelain enamel was covered; in addition, some new features were added.

Receiving special attention this year was the remarkable progress being experienced in new and improved metal preparation operations. Increased use of pickle-free systems for both wet process enameling and for dry powder applications were reported. Statistical process controls were cited for their contribution to quality and cost savings. Advances in the use of powder applications along with innovations in processing equipment received much attention.

A popular part of the program was a question-and-answer, problem-solving period. It proved useful, indicating that we can help each other solve problems if we discuss them and share our experiences.

It is important to stress that each year's Technical Forum program is the product of the efforts of many people. The members of the Technical Forum Committee have contributed freely of their time and talent to produce this year's program. It was a pleasure to work with each member. Making a particularly valuable contribution was my vice chairman Bill McClure. I deeply appreciate his work.

As has been the case since the first Technical Forum back in 1937, the atmosphere of the Forum itself is important. For that, we have our good friends here at the University of Illinois to thank this year. The assistance we have received from the faculty and staff has fulfilled our needs and wishes. We are indebted to Dr. Clifton Bergeron, Dr. David Payne, and to George Conlee of the Department of Ceramic Engineering at Illinois for their help and guidance.

This Forum marks our return to the University of Illinois campus after three years at Ohio State. Next year's Technical Forum will be held at Ohio State on October 7 and 8.

Larry L. Steele, Chairman
1986 Technical Forum Committee
Each issue of Ceramic Engineering and Science Proceedings includes a collection of technical articles in a general area of interest, such as glass, engineering ceramics, and refractories. These articles are of practical value for the ceramic industries. The issues are based on the proceedings of a conference. Both The American Ceramic Society, Inc., and non-Society conferences provide these technical articles. Each issue is organized by an editor who selects and edits material from the conference. There is no other review prior to publication.
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Materials and Processing Developments for Pickle-Free Systems for Wet Process Operations—A Panel Discussion

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Latest developments in materials and processes for the use of pickle-free systems in wet porcelain enameling are reported. Benefits are identified and likely trends in future development work are noted.

Mr. Cook’s Remarks

In 1978, a major appliance manufacturer of home laundry products put into production use a complete cleaned-only metal preparation system for wet application ground coat enamels. Since that time, approximately 24 porcelain enamel plants...
are operating with this type of system. For those not familiar with the cleaned-
only enamel system, metal preparation basically consists of cleaning, rinsing and
a mild neutralizer to control rusting. This cleaning process eliminates the need for
acid etch and nickel deposition. The cleaning process can be with existing immers-
ion or spray wash equipments.

During this 8-yr period, several modifications of the pickle-free frits used at
this home laundry facility were made to overcome various processing problems.
Table I illustrates the steps taken in Chi-Vit laboratory frit development.

Enamel A is the conventional pickle ground coat that was being used at the
time of the conversion to cleaned-only enameling. Enamel B is the first generation
pickle-free ground coat which replaced Enamel A for production use. These first
generation frits were characterized by:

1. Extremely soft glasses.
2. Poor set stability and acid resistance.
3. Prone to saltlining due to solubility.
4. Greater occurrence of ground coat reboil and blistering in cover coat.
5. Very low stress.
6. Poor to no bond when used in combination with cover coat frits.
7. Very good alkali resistance for home laundry parts.
8. Higher cost (12% premium).

Enamel C is representative of the second generation of cleaned-only frits. These
were developed to overcome some of the Enamel B processing problems and were
characterized by:

1. Reduced solubility.
2. Improved set stability and aging characteristics.
4. Lower cover coat rejects due to reboil of the ground coat.
5. Poor acid resistance.
6. Cost premium reduced to 6%.

Enamel D is representative of the third generation of cleaned-only frits
developed for further improvements in production performance and had the follow-
ing characteristics:

1. These new frits could be used as intermediate and hard members in the
frit combination.
2. Solubility and set characteristics were now equivalent to Enamel A.
3. Cover coat rejects were reduced significantly to their old historic levels.
4. Higher stress, pickle-free frits were now available.
5. Because acid resistance was still weak, these frits could only be used in
general purpose or alkali-resistant ground coats.
6. Cost was now almost equivalent to Enamel A.

These series of frit developments bring us to the present day status of what
is new and available in pickle-free frits. Table II lists some of the properties of
six different pyrolytic self-clean enamels developed recently by Chi-Vit laboratories
for some of our range customers.

These fourth generation pickle-free frits used in these enamels are characterized
by the following:

1. They also can be used as intermediate or hard members in the frit
combination.
2. Optimum fire is 804°C (1480°F) or higher.
3. Class A acid resistance or better using P.E.I. Test T-21.
4. Stress of these frits are 400 g and higher.

304
(5) Can be used with 15 to 20% cover coat frits for gray ground coat colors and still retain good bond.

(6) All six of these enamels have completed pickle-free pyrolytic range testing.

(7) Two enamels have been used in production systems for over a yr.

(8) Two are currently being used in a powder production system.

It is apparent that over this time period many improvements have been made in pickle-free frits and it can be said that a pickle-free ground coat system can and is being utilized for almost any type of product.

Mr. Pawlicki’s Remarks

Two yr ago one of my colleagues at Mobay stood before this group and stated “no pickle-no nickel ground coats must be more fluid (at maturing temperatures) in order to develop bond”. As a result of the high fluidity, those frits possessed very narrow firing ranges. Sometimes these ranges were as short as 16 °C (60 °F) as evidenced by the breakdown of bubble structure and the degree of iron oxide penetration into the glass. However, such glasses have gained wide acceptance as evidenced by their popularity. Within the past year new frits have been developed that run counter to the opinions that were considered so important just two yr ago.

Fluidity Measurement

One measurement of fluidity that is employed in the laboratory was earlier described by A. I. Andrews as the “Button” Cylinder Test. This method consists of heating pellets of the enamel frit on a horizontal plane for a given temperature schedule, causing the glass to soften. Then, by means of a suitable device, the panel is altered to the vertical position. Figure 1 shows the results of three no pickle-no nickel ground coat frits along with a standard for purposes of comparison. This test was conducted at 816 °C (1500 °F).

You will note that frits A and B possess relatively long flow lengths which are good indicators of high fluidity. One would expect good bond to result and that is the case. What is surprising is frit C. This frit has a relatively short flow yet it also shows excellent bonding characteristics.

Bubble Structure

Another measure of successful enameling is the bubble structure quality. The bubbles, frozen in the glass, result from high temperature reactions involving frits, clay, mill additions, and the steel substrate. Additionally, grinding, fineness, frit surface tension, and the firing cycle will affect the number and size of the bubbles. When uniformly dense, the bubble structure provides a degree of elasticity. A lack of bubble structure normally reduces the resistance to fishscale defects.

When observed under a microscope at 100X magnification, an opacified bubble structure consisting of very fine bubbles is considered to be underfired. A structure that consists of few bubbles that are large in size is overfired. Through experience it is desirable to have a uniformly dense structure consisting of small, medium, and large bubbles.

Firing Conditions

Enamel slips were prepared for each frit without refractory mill additions. Panels were prepared and fired in three-min schedules that ranged in temperature from 716 °C (1320 °F) to 849 °C (1560 °F) in 4 °C (40 °F) intervals. Cross sections were then mounted in order to properly examine the bubble structures and iron oxide penetration. Figure 2 shows the temperature ranges where frits A, B, and C show good bubble structure, minimal iron oxide penetration, and excellent bond.
Frit A maintained its bubble structure for only 16°C (60°F) extending from 738°C (1360°F) to 771°C (1420°F). A more recently developed product frit B has an extended firing range from 727°C (1340°F) to 804°C (1480°F). At the same time the alkali resistance, as measured by the PEI T-25 procedure, has been significantly improved. Frit C, the short flow product, maintains its bubble structure from 760°C (1400°F) through 827°C (1520°F), thereby providing a product for higher temperature applications.

Summary

It was previously thought that alkali-resistant frits must have high fluidity to ensure proper adherence without pickle and nickel pretreatments. It can now be seen that frits with reduced fluidity such as C can be made sufficiently reactive to develop good adherence in pickle-free applications, yet still have alkali resistance.

In addition, new generation, pickle-free, high-fluidity, alkali-resistant frits have been developed with much improved bubble structures. Furthermore, the alkali resistance of the high-fluidity frits have been improved.

Generally, pickle-free, alkali-resistant ground coat enamels still contain lesser amounts of mill-added refractory than those enamels designed for application over pickled steel due to the detrimental effect on bond. The goal has been to develop frits with all of the desired properties, yet still have the reactivity needed to develop adherence.

Mr. Gazo’s Remarks

Since the last PEI Forum, great strides have been made in the development of pickle-free, wet-pyrolytic ground coat systems. Presently, there are several range manufacturers who have converted to pickle-free pyrolytic systems and others plan to convert. Ferro’s early research and development efforts were concentrated on determining if the present general-purpose or alkali-resistant, pickle-free ground coats would provide adequate pyrolytic properties. These properties were as follows:

(1) Adequate adherence at -1°C (30°F) below optimum fire; excellent adherence at optimum fire and above.
(2) Color stability over firing range ±17°C (±30°F) from optimum.
(3) Heat and craze resistance.
(4) Acid-resistant spot A or better.
(5) Desirable color and gloss.
(6) Cleanability or soil removal.

It became apparent early in our research efforts that the present pickle-free ground coats did not provide the pyrolytic properties. By simply substituting into or modifying existing pyrolytic frit systems, the desirable pyrolytic properties cannot be achieved, particularly heat and craze resistance. What was needed was a totally new generation of pickle-free frits that would provide all of the desirable pyrolytic properties. Since the last Forum, several wet pickle-free systems are in production. Some of the characteristics of these pickle-free pyrolytic systems are:

(1) Colors with L values of 30 or less. This restriction is a result of the high concentration of adherence-promoting frit member needed to achieve excellent adherence.
(2) The mill addition refractory is restricted to 15% or less. Excessive amounts of refractory, such as aluminum oxide, quartz, or titanium dioxide have a detrimental effect on adherence.
(3) The color stability is weaker than the conventional pickle system, but still within production acceptable range.
It does possess excellent refire adherence.

Since the majority of range manufacturers either dip or flow coat the pyrolytic system, a mill addition was developed which provided desirable set and rheology properties, particularly good draining characteristics. It is important that frit solubility be controlled, particularly at the adherence-promoting member. After spending the last two weeks observing the flow coat operation of a pickle-free pyrolytic production system, it is important that plant personnel and supervision need to understand the importance of enamel handling, preparation, and processing. Since the pyrolytic system is a finished or final coat, care should be taken to properly prepare both the enamel and metal substrate.

In order to produce a commercially acceptable pyrolytic oven finish, the enamel slip, metal preparation and processing parameters should be established under plant conditions and followed carefully. With all pickle-free systems, the production ware must be free of heavy rusting and be cleaned thoroughly. The production ware should show no signs of water and be free of drying compound residues. The ware temperature before entering the dip or flow coat area should be as close as possible to the ambient air temperature. Hot or improperly cleaned ware will create instability in the enamel slip; pitting and poor draining will result.

Presently there are several pickle-free pyrolytic systems being used at various range manufacturers. The benefits of converting to the system are numerous. A few of the benefits are: the elimination of the pickle tanks and its associated EPA problems; fuel savings—you no longer have to heat these tanks; the maintenance costs associated with the pickle tanks; and production simplification, to name a few.

Mr. Payne's Remarks

Pickle-free systems have been around for quite a few years now. It can be said that a pickle-free system can be used for almost any type of porcelain product and still maintain product quality. We have been successful in three major ground coat groups (general purpose, acid-resistant, and alkali-resistant). We have been able to use all the existing wet enamel application techniques on all three types of steels available for enameling (cold rolled, which exhibits the best adherence characteristics; enameling iron; and decarburized).

Along with this success has come an accelerated demand for, and development of acid-resistant, pyrolytic, pickle-free ground coats. The demand has become increasingly necessary for those range plants with existing no-pickle general purpose ground coats and no-pickle powder systems.

The pickle-free pyrolytic ground coats have been the most difficult to develop. Contributing to the difficulty is the fact that no-pickle enamels are generally darker because of the addition of adherence oxides, and our restricted use of mill-added opacifiers; this has not been compatible with the fact that range pyrolytics are speckled, and many are light in color with L values greater than 35%. However, range producers have been willing to make concessions on color and frit companies have made improvements in this area as well.

Maintaining thermal durability or craze resistance, and at the same time, retaining bond at below optimum fire has also been a problem. The desire of the enamlers to fire pyrolytic along with cover coats has further complicated the problem. However, with the use of harder frits with the reactivity needed to develop adherence, and the use of lesser amounts of refractory materials as mill additions, we have been able to achieve a satisfactory balance.

It should be noted that since the bonding mechanism is built into the frit, adequate firing is necessary to promote adherence, as it is in all pickle-free ground
coats. This is especially true of pyrolytics, since softer, more fluid bonding frits cannot be used in some cases. Making sure parts to be enameled are free of cleaner residues as well as oils, dirt, scale, and rust has also become an even more important consideration of the enameler, especially those with immersion systems.

Technology for the pickle-free pyrolytics has advanced substantially over the past year. Frit companies have done much to improve the quality of the enamels in production to date with regard to chemical resistance, thermal durability, color, color stability and bond. Also, enamblers have now had another year of production experience. Since last year, pickle-free pyrolytics have become a way of life for many; many more will follow in the future.

We as a company realize that there is still room for improvement and we will continue to work toward making improvements in the coming year.

Table I. Laboratory Frit Development

<table>
<thead>
<tr>
<th>Enamel</th>
<th>Fusion flow mm</th>
<th>Stress g</th>
<th>Optimum temp °C</th>
<th>Cost*</th>
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<tr>
<td>A</td>
<td>77</td>
<td>274</td>
<td>788 1450</td>
<td>Std</td>
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<tr>
<td>(Previous production)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>87</td>
<td>237</td>
<td>771 1420</td>
<td>+12%</td>
</tr>
<tr>
<td>(1st Generation pickle-free)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>90</td>
<td>229</td>
<td>771 1420</td>
<td>+6.1%</td>
</tr>
<tr>
<td>(2nd Generation pickle-free)</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>D</td>
<td>78</td>
<td>275</td>
<td>785 1445</td>
<td>+1.7%</td>
</tr>
<tr>
<td>(3rd Generation pickle-free)</td>
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*Cost premium as a percent of Enamel A.

Table II. Properties of Pyrolytic Self-Clean Enamels

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<tr>
<th>Enamel</th>
<th>Fusion flow mm</th>
<th>Stress g</th>
<th>Optimum temp °C</th>
<th>Optimum temp °F</th>
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<tr>
<td>I</td>
<td>60</td>
<td>398</td>
<td>804 1480</td>
<td></td>
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<tr>
<td>II</td>
<td>59</td>
<td>364</td>
<td>807 1485</td>
<td></td>
</tr>
<tr>
<td>III</td>
<td>53</td>
<td>442</td>
<td>818 1505</td>
<td></td>
</tr>
<tr>
<td>IV</td>
<td>51</td>
<td>437</td>
<td>821 1510</td>
<td></td>
</tr>
<tr>
<td>V</td>
<td>62</td>
<td>346</td>
<td>804 1480</td>
<td></td>
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<tr>
<td>VI</td>
<td>59</td>
<td>420</td>
<td>807 1485</td>
<td></td>
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</table>
Fig. 1. Results of button cylinder test on three no-pickle no-nickel ground coat frits along with a standard for purposes of comparison.

Fig. 2. Firing range of three no-pickle no-nickel ground coat frits as a function of bubble structure.
Production Experiences With Pickle-Free Systems for Wet Process Operations—A Panel Discussion

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The use of pickle-free systems in production is described. Observations are reported on steel, metal preparation, and firing as they affect pickle-free systems.

Mr. Sedalia’s Remarks

A few years ago when the Environmental Protection Agency promulgated the wastewater regulations affecting the porcelain enameling industry’s pickling process; we had no choice but to comply.

The excessive discharging of iron and nickel into POTW was the major problem. The discharging of iron was decided to be regulated by converting regular sulphuric acid tank into oxy-acid because oxy-acid tank is rarely dumped, and the amount of iron discharged during the daily dumping of acid rinse tanks was posing no problem because it was meeting EPA’s guidelines. But for controlling nickel discharge, we had three options:

1. To change the enamel system which requires nickel deposition on steel compared to that which does not require nickel deposition on steel for obtaining bond. Cost: $0.20 more per product.

2. To keep the low cobalt ground coat enamel in production and build a wastewater treatment facility. Cost: $350 000 for treatment facility and $40 to $60 000 yr for operation and maintenance.

Narayan M. (Nick) Sedalia is a materials engineer at Emerson Contract Div. of Emerson Electric Co. Prior to his present position, he was a ceramic engineer at Hobart Corp. He holds a B.S. in chemistry from the Univ. of Bombay and a B.S. and M.S. in ceramic engineering from the Univ. of Missouri at Rolla.

Floyd J. Williams is manager of enamel engineering at Porcelain Metals Corp. He began work with the company in 1983 and was district sales manager for 30 yr at Mobay Chemical Corp. before assuming his present position. He received his B.S. and M.S. in ceramic engineering at Ohio State Univ.

Daniel H. Luehrs is currently finishing superintendent at Magic Chef where he has been involved in the company’s new powder line. He is a ceramic engineering product of Rutgers University and before joining Magic Chef, worked for both Whirlpool and Crane Plumbing.
To keep the low cobalt ground coat in production and capture the overflow of nickel rinse solution into a nickel tank at the evaporation rate of nickel solution. When the nickel rinse tank has excessive nickel or gets contaminated, haul the nickel rinse solution elsewhere as a hazardous waste. Cost: A. Hauling cost—$2000 to $3000/haul—one or twice a mo. B. Take a risk of nickel rinse solution spillage in plant, in transit or at dump site.

Considering these factors, option number one was selected. Now, the nickel tank has been converted into another acid rinse tank and the nickel rinse tank into another neutralizer tank.

You may wonder why we need an oxy-acid tank with a pickle-free type ground coat enamel? Our product has over 0.3 m (11 ft) of welded seam and we want to be certain that we achieve excellent bond at that area and maintain that quality of bond up to at least five firing cycles. The oxy-acid tank is operated at 66°-71 °C (150°-160°F) with about 1% ferric level. The immersion of dishwasher tub for three to four min adequately removes the weld scales. The removal of weld scale not only provides excellent bond but also assures against enamel pop-off. However, we are still working on eliminating the oxy-acid pickle system and on using a clean-only system.

The processing of no nickel or pickle-free ground coat enamel is very critical. This enamel system is least forgiving; thus it does not leave much room for processing errors. However, if the processing steps are followed within the established guidelines, the product will have consistent quality day after day.

We at Emerson are committed to produce the same top-notch KitchenAid dishwashers as we did under KitchenAid management, and the same quality will be delivered to our other dishwasher customers.

**Mr. Williams’ Remarks**

We started a program in 1984 to get away from the traditional metal preparation of acid etch and nickel deposition. We have been able to control the nickel in our water effluent to meet state and federal limits but we wanted to shake the dependence on nickel in our metal pretreatment.

We solicited proposals from the frit suppliers for a ground coat system which would be applied over a clean metal surface without acid etch or nickel. We tested several formulations and picked two frit systems which showed promise over clean-only steel.

We looked at the following parameters:

1. Frit ratio—Vary the amount of the primary adherence frit in the mill. Develop a formula for optimum bond and fired surface.
2. Clay addition—Vary the clays to improve bond and workability.
3. Refractory—Determine the optimum level of silica or other refractory which will not decrease bond.
4. Mill additions—Check additions of oxides, opacifiers, and salts and their effect on bond. Also, additions of adherence promoting agents, such as nickel oxide and antimony oxide.
5. Cost—Cost variance from our regular ground coat system.

When we were satisfied with the formulation of the pickle-free ground coat systems, we started with small production mills of each frit system to check production parameters.

1. Temperature range—Check for adherence and resistance to overfire and repeat fires. Look for burnoff and copperheads. We found that the minimum temperature for adherence is closer to optimum firing temperature.
Refire performance—Does a poor adherence on first fire develop better adherence on refire? Look again for copperheads.

Adherence under cover coat—Does cover coat application adversely affect the ground coat adherence?

Adherence on various metals—Make sure that the ground coat will react well with enameling iron, decarburized steel, and cold rolled.

We repeated the small production mills several times to make sure that the results would be reproducible. We also adjusted the formulation to give us the desired set consistency for production conditions.

Then we arranged with our customers to test the ground coat systems on their parts and give us approval to supply their parts with the new pickle-free ground coat system. This approval was received late in 1985. We made the change to pickle-free ground coat on most parts in January of this year and have run consistently good enamel parts comparable to our regular ground coat with pickle.

Our experience has underlined process variables which must be controlled.

1) Metal preparation—Make sure that the metal surface is uniformly clean and free of oxide film from corrosion or welding. A light bloom or red rust should not be a problem. The pickle-free ground coat will be more reactive over pickled steel and will tend to overfire more easily. Try not to mix parts with different metal preparation when using a pickle-free ground coat system. We have adopted a CR (cleanliness rating) test for clean only pretreatment. The test gives us a confidence in providing a uniformly clean metal surface for enameling. (Dave Mimms will have more to say on this test later in the program.)

2) Firing temperature—Determine the optimum temperature for the pickle-free ground coat system and make sure that the furnace maintains this temperature. Work to eliminate any cold spots or temperature variations within the furnace. Maintain a daily temperature record and check production parts each day to verify the adherence of the ground coat. Remember you don’t have as much leeway with a pickle-free ground coat system. But with attention to details, you can eliminate your acid and nickel pretreatment and produce good quality porcelain enamel parts.

Mr. Luehrs’ Remarks

I would like to take just a few minutes today to continue with some observations on the benefits that we’ve seen with ground coat and pyrolytic wet pickle-free enamels at Magic Chef at our Cleveland, TN plant. Most of the benefits that you realize with pickle-free systems are in the metal preparation area. Working towards eliminating the acid and the nickel batch immersion systems leads us to what we’ve done in the plant in replacing these immersion systems with modern, clean-only spray washers. These spray washers, basically, are a series of counter flowing alkaline cleaning stages, followed by rinse stages. We don’t use currently in the plant any neutralizers at all. We go directly from the clean to the rinse, into the dryers, and into the process applications. One of the first advantages we saw with the systems was a great reduction in chemical costs. There is an enormous cost in the acid, the oxi-acid materials, the nickel costs and we also saw a reduction in costs in the cleaner usage itself. But, probably the biggest advantage that we have seen throughout the years since we have gone to pickle-free, is the reduced burden on the waste treatment facility. The waste treatment solid loads have been significantly lowered. We have even begun sending some waste water directly to the city for their handling. The resulting metal preparation systems that we are using in Cleveland are very simple to maintain; we no longer have to deal with the nickel control and the acid/oxi-acid controls. We are simply doing temperature...