Problems with Surface Protection of Welded Joints by Vitreous Enamel Coating

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Abstract. This work deals with the application of a vitreous enamel coating on welded joints, whereas vitreous enamel being intentionally applied in high thicknesses. As a base material, we used low-carbon steel Kosmalt E 300 T. Two different processes were chosen for pre-treatment - chemical and mechanical. The resulting enamel coating consisted of two successively applied layers of base and cover enamel coating. The thickness and microhardness of the coating were measured. The metallographic analysis was also performed on selected samples. The results show that the high thickness of the enamel coating significantly impairs the useful properties of the product.

1 Introduction
The basic raw material for enamel production is an enamel frit (vitreous glass granules). It is produced by melting a mixture of various materials, such as silica, borax, potassium chloride, alumina, titanium dioxide, and other metal oxides. Enamel frit is a key ingredient for making enamel slip which is a mixture that is suitable for applying to a metal surface. Enamel slip is created by milling enamel frit with water, clays, pigments, and electrolytes into wet suspension in a ball mill that contains porcelain-grinding materials [1].

The main reason for the wide use of enamel coatings in the industry is their corrosion resistance. The enamel coatings also have great resistance to mechanical abrasion and chemical resistance. Due to their glassy nature coatings exhibit excellent impermeability, washability, high-temperature resistance, and fire resistance. Another advantage of enamel coating is its ability to withstand UV radiation with the characteristics of the surface unchanged in time [2]. This feature together with wear and chemical resistance makes enamel coatings suitable for the pharmaceutical, food, or chemical industry.

In these industries, there are cases where it is necessary to form an enamel coating on a product that contains welded joints, such as pressure vessels for various chemicals or water heaters. The welded joint will create chemical and geometric inhomogeneity on the surface of the product. One problem is the strong increase in coating thickness around the weld bead. The recommended total maximum thickness of the enamel coating for consumer goods is 450 microns according to [3]. For use in aggressive environments, it is permitted to create a higher thickness, but in the vicinity of the weld bead, the thickness of the coating may increase several times. Another problem is chemical inhomogeneity in the area of the welded joint.
The presented work focuses on the application of a vitreous enamel coating of high thicknesses. In the experimental part of this work, very thick coatings were intentionally created, corresponding to the geometric shape of the weld bead. In the extreme case, the thickness of the enamel coating reached almost 1500 microns.

**Experimental**

1.1 *Base material*

As the base material for welding was used low carbon steel Kosmalt E 300 T. Table 1 shows the chemical composition of the base material as well as the weld metal. Glow-discharge optical emission spectrometry (GDOES) analysis was performed using a Spectrum Analytic optical emission spectrometer according to [4, 5].

| Element | C   | Mn | Si  | P   | S   | Al  | Cu  | Ti  |
|---------|-----|----|-----|-----|-----|-----|-----|-----|
| Base Metal | 0.039 | 0.195 | 0.027 | 0.013 | 0.011 | 0.048 | 0.021 | 0.060 |
| Weld Metal | 0.064 | 0.942 | 0.605 | 0.011 | 0.014 | 0.009 | 0.026 | 0.007 |

1.2 *Preparation of samples*

Samples for welding were prepared by cutting from a whole piece of Kosmalt E 300T steel sheet with a thickness of 5 mm. The individual samples had dimensions of approximately 198x90 mm. Before welding, the edges of the weld surface were chamfered to 30° and stitched. The ABB IRB 1660 ID-4 welding robot was used for welding. Method 135 (MIG / MAG) was used for all samples. A solid wire from ESAB with the designation OK AristoRod 12.50 (C = 0.10 %, Mn = 1.50 %, Si = 0.90 %) was used as additional material. The size of the weld gap ranged from 1.0 to 1.6 mm.

The welding current ranged from 210 to 250 A and the welding voltage from 18 to 24 V. Shielding gas M21 (82 % Ar + 18 % CO₂) with a flow rate of 12 l/min was used to protect the weld according to the standard EN ISO 14 175. The welding speed was 5 mm/s.

1.3 *Pretreatment*

For the first set of samples (K-01, R-01A, R-02A, R-03A, and R-04A), the pretreatment consisted of the following steps. Degreasing by an aqueous solution of Simple Green in ratio 1:15 at room temperature (approximately 20 °C) for 20 minutes. Then samples were pickled in 10 % HCl solution at 27 °C for 18 minutes. Following neutralization was carried out in 10 % NaOH solution at 40 °C for 4 minutes. Finally, samples were rinsed with demineralized water and dried in a drying oven at 100 °C.

The pretreatment of the second set of samples (K-02, R-01B, R-02B, R-03B, and R-04B) consisted of blasting the surface after welding. Silica sand was used as the blasting material. Before enameling, all samples were degreased in an aqueous solution of Simple Green in a ratio of 1:15 for 20 minutes. Then rinsed with demineralized water and dried in a drying oven at 100 °C.

1.4 *Enameling*

The enamel suspension was applied by the wet method. Both the base and cover enamel layers were applied by hand spraying with a pressure gun.

After application and drying the blue Ferro base enamel, the samples were placed into a furnace and fired at 840 °C for 12 minutes. The cover green Mefrit enamel was applied after samples cooled down to room temperature and then dried and fired at 830 °C in the furnace for 9 minutes burning times and temperatures were determined based on previous experience. Samples R-02A and R-02B were selected for further analysis.
2 Results

2.1 Thickness measurement

Elcometer 456 digital thickness gauge was used to measure the thickness of the enamel coating layer. The measurement was performed according to the ISO 2178 standard. The samples were divided into 3 measuring areas - above the weld (Area 1), in the weld area (Area 2) and below the weld (Area 3), see Figure 1. In each area, fifty measurements were performed.

The measured thickness of the enamel coating in Area 1 ranged from 775 µm to 1498 µm. In Area 2 it ranged from 718 µm to 1495 µm. The coating thicknesses ranging from 556 µm to 1478 µm were measured in Area 3. Summarized measured coating thicknesses are listed in Table 2.

![Figure 1: Measuring areas – Area 1, Area 2, Area 3.](image)

| Sample | Area 1 [µm] | Area 2 [µm] | Area 3 [µm] |
|--------|-------------|-------------|-------------|
|        | Mean  | Std. Dev. | Mean  | Std. Dev. | Mean  | Std. Dev. |
| K-01   | 1391  | 76       | 1362  | 93       | 1271  | 107       |
| R-02 A | 893   | 97       | 830   | 69       | 610   | 31        |
| K-02   | 1210  | 66       | 1395  | 62       | 1015  | 64        |
| R-02 B | 1404  | 44       | 1359  | 63       | 861   | 83        |

![Figure 2. Comparison of mean values of measured thicknesses in three measured areas.](image)

Figure 2 shows a comparison of the measured thickness of the enamel coating on selected prepared samples. The coating thickness on the R-02A sample reached the lowest values in all measured areas. Unlike the other samples, the enamel coating of sample R-02A did not peel off in preparation for further analysis. When comparing the thicknesses, it is clear the peeling occurred due to higher values of the thickness of the enamel coating.
2.2 Microhardness measurement

The microhardness measurement according to Vickers was performed following the EN ISO 4516 standard. The microhardness was measured on a LECO microhardness tester LM 247 AT with a load of 100 g. Eight indentations were made in one line, out of the weld area. Table 3 shows the comparison of microhardness measured values of R-02A and R-02B samples.

According to the paper [6], the microhardness of the enamel is, inter alia, dependent on the temperature and the firing time. The measured values correspond to the microhardness given in the [6] work and, after conversion to megapascals, also partially correspond to the results obtained in the [7] work.

| Table 3. Microhardness measurement, HV$_{0.1}$ |
|-----------------|---|---|---|---|---|---|---|---|
|                | 1  | 2  | 3  | 4  | 5  | 6  | 7  | 8  | Mean | Std. Dev. |
| **R-02A**      |    |    |    |    |    |    |    |    |       |          |
| Base layer     | 573| 594| 585| 578| 551| 595| 546| 597| 577   | 20        |
| Cover layer    | 632| 621| 608| 593| 610| 626| 579| 640| 614   | 20        |
| **R-02B**      |    |    |    |    |    |    |    |    |       |          |
| Base layer     | 568| 607| 573| 568| 612| 627| 646| 648| 606   | 33        |
| Cover layer    | 646| 676| 738| 570| 618| 570| 698| 521| 630   | 74        |

The sample R-02A (pretreated by pickling) was measured without more frequent deviations. Compared to that the coating on sample R-02B pretreated by blasting contained a larger number of pores and was, therefore, more difficult to measure. The indentor often penetrated directly into the pore. The increased number of pores in the coating of sample R-02B could be caused by different pretreatment.

1.3 Metallographic analysis

For better visualization of the enamel coating on the welded joints were samples subjected to metallographic analysis. The samples were cut and encapsulated. Prepared capsules were subsequently mechanically treated by grinding and polishing. To highlight the resulting structure, the samples in the capsules were etched with Nital etchant. Figure 6 shows a split sample R-02A.

![Figure 6. View of the split sample R-02A.](image)

Figures 7 and 9 show the macrostructure of the cross-section of samples R-02A and R-02B, respectively. There is a clear difference in the thickness of the coating on both samples. The cross-section of sample R-02A (Figure 7) clearly shows the individual layers of the enamel coating. The figure shows the thicknesses of the base and cover enamels, the base enamel ranged from 237 to 588 microns, the cover enamel ranged from 205 to 269 microns. The basic layer of enamel is blue and the covering layer of enamel is green.
Figure 7. Macrostructure of the sample R-02A.

Figure 8 shows divided sample R-02B on which the enamel peeled off during cutting. It is clear the thickness of the layer significantly affects the adhesion of the coating.

Figure 8. Divided sample R-02B.

The cross-section of sample R-02B (Figure 9) clearly shows the individual layers of the enamel coating. The figure shows the thicknesses of the base and cover enamels, the base enamel ranged from 488 to 1175 microns, the cover enamel ranged from 376 to 471 microns. The basic layer of enamel is blue and the covering layer of enamel is green.

Figure 9. Macrostructure of the sample R-02B.

4 Conclusion

This study deals with the application of high thickness vitreous enamel coating on welded joints. Very thick coatings were created within the experiment, corresponding to the geometric shape of the weld bead. The lowest measured thickness of the enamel coating was 610 ± 31 µm on the sample R02-A. The highest measured thickness of the enamel coating was 1404 ± 44 µm on the sample R02-B. The cutting process of the samples confirmed that a higher thickness of the enamel leads to greater destruction of enamel coating. For samples with higher thickness of the coating the enamel peeled off during cutting.

Samples R-02A and R-02B which differed used pretreatment before application of enamel coating were used for further research. Both samples included pores of different amounts and sizes. The presence of pores was higher in the sample R-02B.
Furthermore, the microhardness of the base and cover layer of the enamel was measured by the Vickers method with a load of 100 g. The mean microhardness of the base layer of the sample R-02A was 577 HV0.1 and the mean microhardness of the cover layer of the same sample was 614 HV0.1. The mean microhardness of the base layer of the sample R-02B was 606 HV0.1 and the mean microhardness of the cover layer of the same sample was 630 HV0.1. The study shows that the coating thickness should be uniform and without major fluctuations. This could be achieved either by high-quality spraying (requires good skills of the person) or, at best, by applying powder enamel in a high-voltage electrostatic field [8]. In addition, this technology is not so sensitive to the quality of the surface before enameling (pretreatment) [8]. The practical results of powder enameling in a high-voltage electrostatic field have been confirmed on production lines in the production of steel bathtubs [9], or in the production of welded boilers for hot water preparation [10]. It is clear the higher thickness of the coating significantly worsens the adhesion of the coating. From the point of view of welding technology, the use of low-carbon welding wire can be recommended, as the increased carbon in the weld metal can increase the formation of pores in the enamel coating [2]. For the same reason, it would be appropriate to use an inert gas (pure argon) instead of a mixed gas (Ar-CO2). Of course, it would be beneficial to mill the weld to the plane of the base metal, this variant will be discussed in the next study.

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