ENERGY-TO-FAILURE TO ASSESS THE QUALITY OF ELECTRO FUSION JOINTS IN PE PIPES

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ABSTRACT

When electro fused joints in newly installed PE100 pipelines are tested using the Peel test (ISO 13954 [1]) they too often fail in a brittle manner. Contamination in the interface between the surfaces to be fused is usually the cause. About 77 % of the contaminants in 39 cases of brittle test bars could be identified by chemical analysis. Silicones were found in 23 % of the cases. In 54 % of the cases inorganic particles consisting of silicate (sand, clay) or calcium carbonate (from cement dust) had been trapped by electrostatic charges on newly-skinned PE pipe surfaces. Fine particles pose a larger threat to joint quality than coarse particles. In 13 % of the cases the presence of sweat, hand balm and detergent was suspected. In 15 % of the cases the identification was inconclusive.

By integrating the area under the force-displacement curve in the Peel test, the total amount of energy needed to produce failure was determined. This Energy-to-Failure (EtF) method delivers quantitative results and opens up new possibilities for studying correlations with important jointing variables and with external influences, such as contamination of the joint surface.

Commercial cleaning liquids developed to remove silicone contamination before jointing were tested and compared to ethanol using the EtF method. While ethanol is partially effective, the silicone removers are more effective, although they evaporate more slowly. For the least contaminated joints their quality is not only higher but also depends on the gap between pipe surface and coupler surface and therefore on pipe ovality.

Using isopropanol instead of ethanol for cleaning pipe surfaces may lead to the formation of voids. This occurs when the time allowed for drying is not prolonged. The lower volatility and higher solubility in PE of isopropanol lead to more absorption in the skinned outer pipe surface. At the high fusion temperature bubbles filled with isopropanol vapour are locked in by the molten PE, which subsequently remain as highly undesired voids after cooling. GC-MS proves that isopropanol is still present in the voids and is therefore responsible for their formation. A return to ethanol as cleaning liquid removes such voiding.

INTRODUCTION

The integrity of pipelines in which heat-fused joints have been used must be very high. This is necessary in order to ensure the safety of gas pipelines. After closing the trench, it is no longer possible to assess joint quality. This has led some of the sponsors of the present investigation (see Acknowledgements) to remove a certain percentage of joints from newly-installed PE100 pipelines and have these tested using the Peel De-cohesion test, also denoted the Peel test [1]. A certain percentage of such joints do not meet the requirements of EN 1555. It was assessed previously [2] that in case a fusion plane fails brittle, it often contains impurities. Other investigators have also published on contaminations of pipe surfaces and how these influence joint quality [3-8]. Marshal et al [3] explain why finer silicate particles are more detrimental to joint quality than coarser particles.

The goal of the present investigation is to determine the most important and abundant contaminants that occur in practice and determine their origin in order to reduce such contaminations in electro fused joints in future.

MATERIALS AND METHODS

Part of the work was performed on newly-produced electro fused joints from PE100 gas pipelines. A certain percentage of such joints was removed from the pipeline and tested in the laboratory using the Peel test. A certain percentage of these joints proved to be brittle and hence failed this test.

Brittle joint planes were investigated using Infrared Reflectance Spectroscopy (IRRS) or Scanning Electron Microscopy (SEM) annex X-Ray Micro Analysis (XMA).
The “information depth”, i.e. the depth into the surface from which chemical information can be collected, is 2-5 microns for IRRS and about 1 micron for XMA.
In some cases Gas Chromatography with Mass Spectrometric detection (GC/MS) was also performed.
Additional joints between PE100 pipes were produced in the laboratory. All skinned pipe surfaces were cleaned using Tangit ® ethanol from Henkel. Other commercial cleaning liquids intended for silicone removal were also used, denoted Cleaner 1 and Cleaner 2.

RESULTS

The results are divided into four parts.
The first investigation was performed in order to assess the type and origin of contaminants in joints that had failed in a brittle manner in the Peel test. During this investigation poor joint quality caused by jointing mistakes in the field was excluded. The second part consisted of assessing whether commercial cleaning agents can be used to remove one particular type of contamination, silicone spray. In the course of this investigation the third part was undertaken: development of the new method of determining the Energy to Failure (EtF), also denoted the Failure Energy, during the Peel test. The fourth part consisted of results obtained with a less volatile cleaning agent, isopropanol, also denoted isopropyl alcohol (IPA).

1. Contaminants in joints from practice

Failure modes of electro fused joints encountered after the Peel test that meet the requirements of EN 1555 are IP (failure in the pipe wall – Figure 1) and BW (ductile failure between the heating wires – Figure 2).

Figure 1. Failure mode IP (in the pipe wall) after the Peel test.

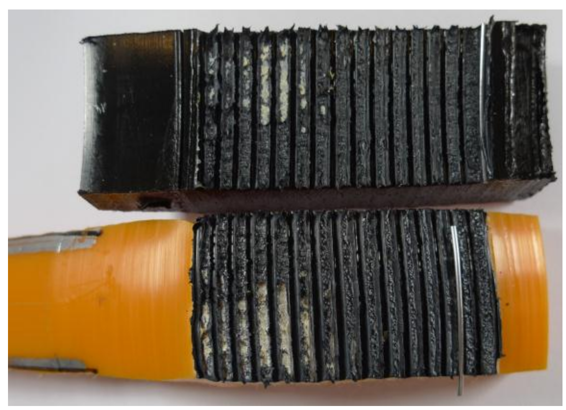


Figure 2. Ductile failure between the heating wires (BW) after the Peel test. Black PE from the fitting still adheres to the orange pipe.

However there is a third failure mode that is not acceptable: condition BF (brittle failure - Figure 3).
In this case there is no adhesion of the black fitting material to the external surface of the pipe. Such brittle joints, 39 in total, were investigated using the techniques mentioned in the previous section.

Figure 4 shows infrared spectra measured using IRRS of a brittle joint plane and of an uncontaminated reference sample. Whilst the majority of the peaks are due to Polyethylene and its normal additives, differences are only noted at around 1600 and in the “valley” around 1400 cm^{-1}. Therefore, a difference spectrum is needed and the red spectrum is subtracted from the blue one.
This difference spectrum is shown in Figure 5, together with the best match (although not a perfect one) from the Biorad/Sadtler database (KnowItAll ®, containing 220,000 reference spectra), which is sodium lactate. Sodium chloride and lactic acid are the most prominent components in human sweat. XMA was not performed on this sample.

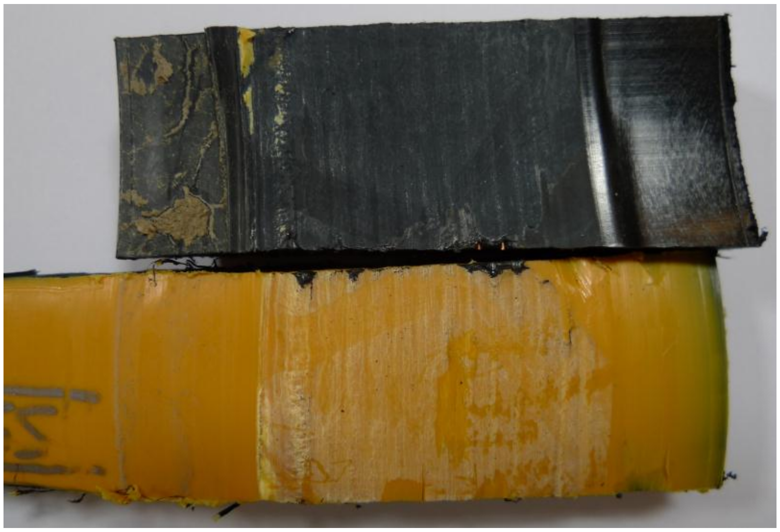


Figure 3. Brittle failure of a test bar after the Peel test. There is no adherence between the black PE material in the fitting and the orange pipe.

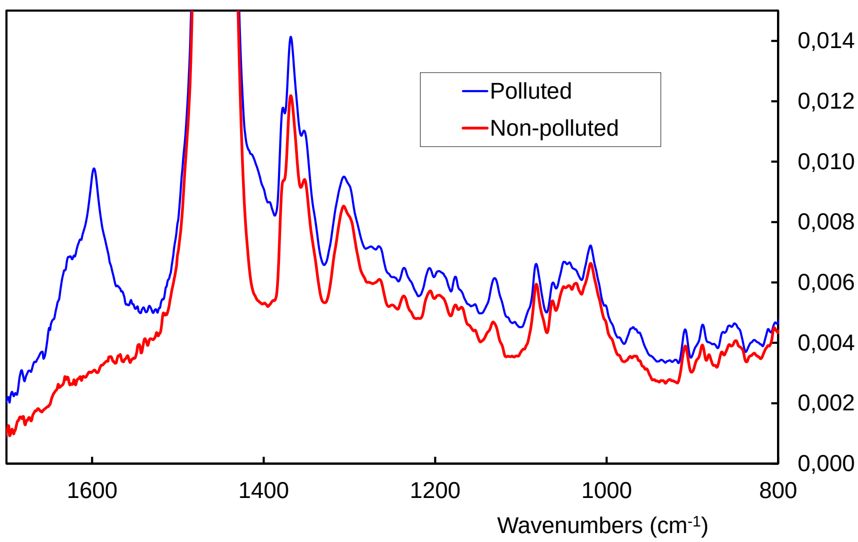
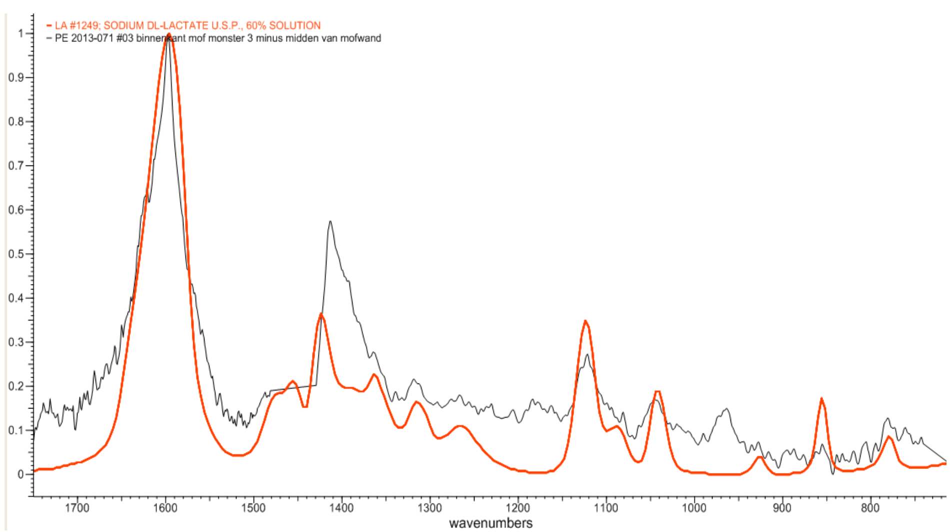


Figure 4. Infrared spectrum of a brittle joint plane (blue) and of a reference sample cut from the middle of the wall of the same pipe (red).

Figure 5. Black: difference spectrum (blue spectrum minus red spectrum in Figure 4). Between 1480 and 1430 cm^{-1} a poorly compensated PE peak was replaced by a straight line to improve spectral searching. Red: the best match among the reference spectra in KnowItAll ® is sodium lactate.



An interesting result is presented in Figure 6. SEM-XMA performed on another brittle joint plane reveals that sodium chloride crystals are arranged in concentric circles. This suggests that a drop of human sweat had splashed onto the pipe surface before fusion had started.
In the previous sample which probably contained sodium lactate (Figure 5) and which was only analysed using IRRS, no traces of sodium chloride were found, because this substance has no infrared spectrum.

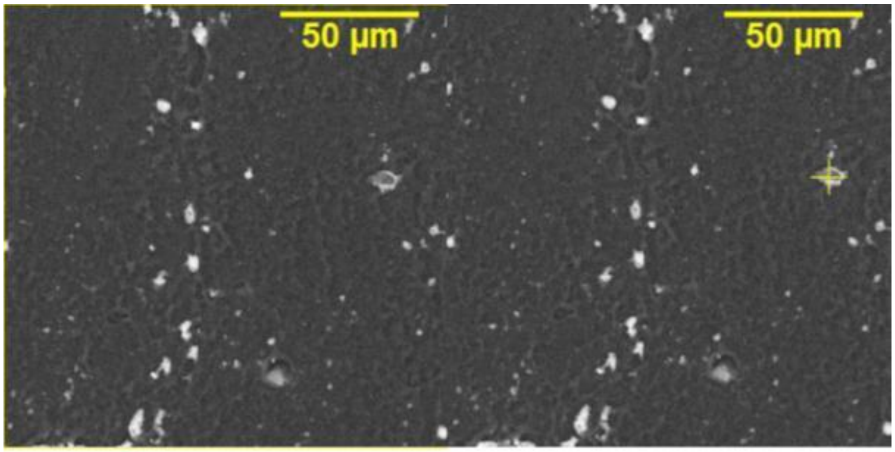
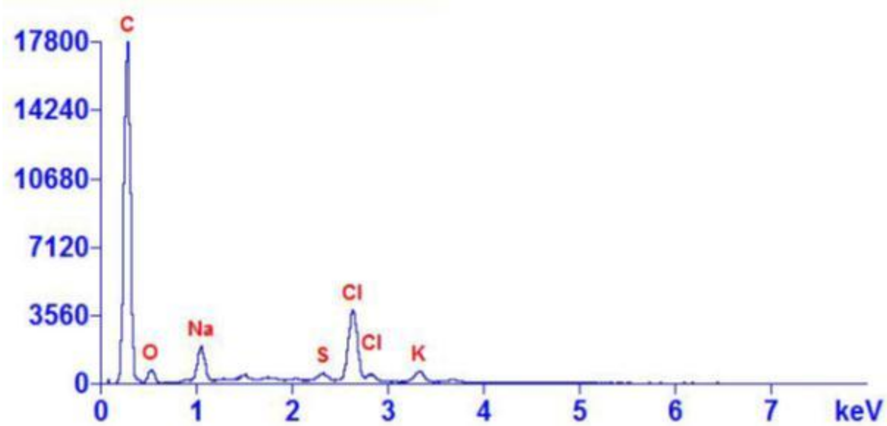


Figure 6.
a. SEM image showing concentric circles of white crystals on a brittle joint plane.



b. The EDX spectrum shows that the crystals consist of sodium chloride.

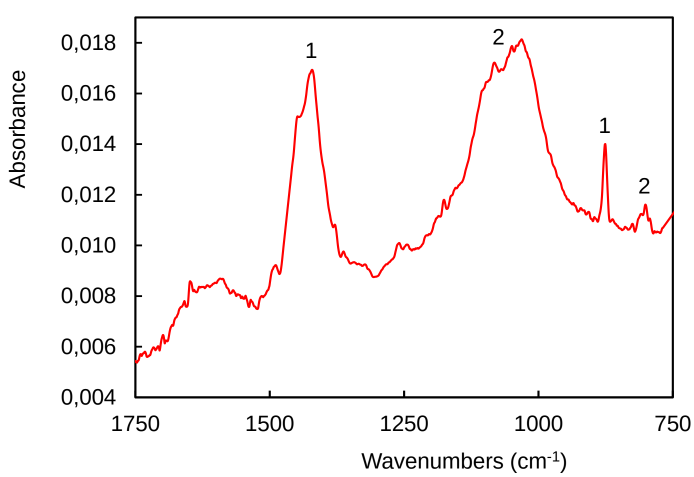


Figure 7. Infrared difference spectrum: spectrum of a joint area that failed in a brittle manner in the Peel test minus the spectrum of uncontaminated PE. By the subtraction the large peaks of PE are compensated. Contamination with calcium carbonate (peaks 1) and silicates (peaks 2) are found.

IRRS performed on in another brittle joint plane reveals the presence of two different contaminants, silicate and calcium carbonate (Figure 7). Both substances show two clear bands and this provides definite identification. Other important contaminants such as silicones were noted in many cases. Detection of silicones and silicates has been described previously [2].

2. *Cleaning agents for silicones*

Silicones are an abundant contaminant. Commercial cleaning agents meant for removing it (Cleaner 1 and Cleaner 2) were tested (Table 1). An uncontaminated pipe was skinned using a rotating device and cleaned using Tangit ethanol (condition Ref). Next the skinned surface of such a pipe was contaminated with silicone spray (condition S). The following three cleaning conditions were all based on condition S.

Table 1. Conditions for electro fusion jointing.

Code	Condition	Code	Cleaning condition
Ref	Uncontaminated joint	SE	as S, after cleaning with ethanol
S	Pipe contaminated with silicone spray	SC1	as S, after cleaning with Cleaner 1
		SC2	as S, after cleaning with Cleaner 2

Three cleaning agents were used: ethanol, Cleaner 1 and Cleaner 2. In all cases the contaminated pipe surface was cleaned twice.

Initially, the traditional manner of evaluating the Peel test results was used, i.e. visual observation of the failure mode. The failure modes are presented in column 3 and 4 in Table 2. In all conditions except the contaminated condition S, the joint mainly fails in the pipe (IP). Condition S mainly leads to ductile failure between the wires (BW). This result is also acceptable. Condition BF does not occur in this series of lab-produced joints. Conditions BW and IP always add up to 100 %. Therefore, all joints evaluated in Table 2 meet the requirements of EN 1555. Given the visual observations, this outcome is not satisfactory.

Table 2. Peel tests at 25 mm/minute on electro fused joints in 110 mm SDR11 pipes that had been contaminated with silicone spray (S) and cleaned afterwards using ethanol (SE), Cleaner 1 (SC1) and Cleaner 2 (SC2). Ref: non-contaminated joint. Between parenthesis: standard deviation.

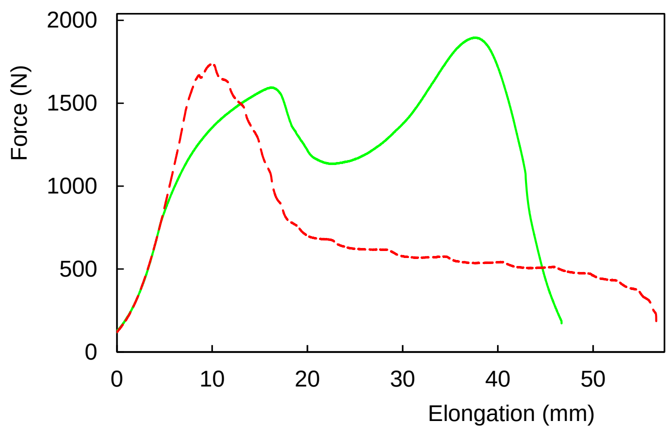
Series	Number of test bars (n)	Failure type BW (%)	Failure type IP (%)	Maximum Force (N)	Elongation at Break (%)
Ref	12	8	92	66,2 (19,5)	36,1 (11,3)
S	12	75	25	64,6 (5,1)	33,0 (4,9)
SE	12	25	75	60,7 (6,2)	33,6 (5,0)
SC1	11	18	82	60,6 (7,0)	37,3 (11,8)
SC2	11	18	82	65,7 (8,5)	35,4 (7,2)

BW: Ductile failure between the wires.
IP: Failure in the pipe.

A second evaluation method is based on the force-displacement curve measured during the Peel test. Usually these curves are not recorded, but they are informative. Figure 8 shows two typical examples. From these curves the maximum force and elongation were determined and used as a quality criterion (columns 5 and 6 in Table 2).

It is obvious that this evaluation method still does not allow a clear differentiation between the various cleaning conditions and that the standard deviations are too large. Therefore, yet another parameter is needed. This is presented in section 3.

Figure 8. Typical examples of the force versus the displacement of peel test bars. The Failure Energy (Joule/mm) is the area under the curve (Joule), divided by the width of the test bar (mm).



3. <u>Energy-to-Failure (EtF) or Failure Energy method</u>

The EtF method uses the area under the force-displacement curve in Figure 8. At each separate data point of the curve, the force (N) is multiplied by the displacement (mm), allowing the energy in Nm or Joules to be calculated. Since ISO 13954 specifies that the width of the test bar should lie between 25 and 30 mm, some variation in width between different test bars occurs. Therefore, the EtF method specifies the mechanical energy taken up until failure in Joules per mm width of the test bar.
The EtF was determined from the already measured force-displacement curves. The average EtF per condition is given in Figure 9. There is some indication that the thicker wall of the 200 mm pipe (condition R200) leads to a higher EtF value.

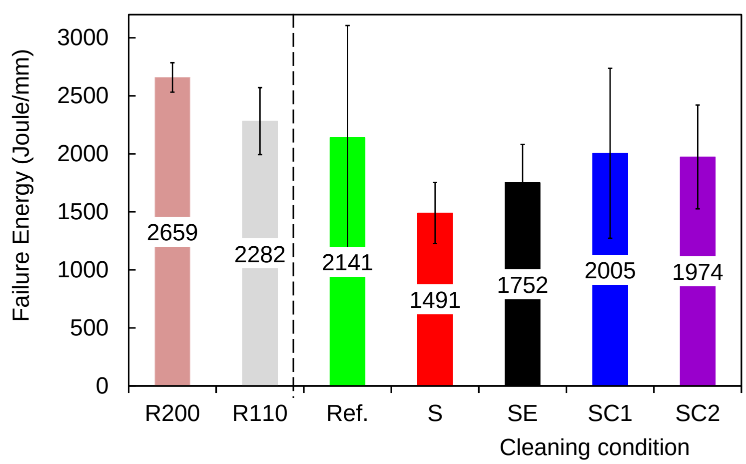


Figure 9. Failure Energy (Joule/mm) for different contamination and cleaning conditions. R200 and R110 are non-contaminated reference joints from another investigation which give similar results to Ref. Average value per condition given at half height of each column.

The Failure Energy of the test bars contaminated with silicone spray (S) is lowest with respect to condition Ref. Cleaning with ethanol provides some improvement. However, the commercial silicone cleaners perform best. There is nevertheless much scatter in some of the data.

This scatter can be partially explained by the influence of the clearance between the outer surface of the pipe and the inner surface of the fitting. This clearance, which is caused by ovality of the pipes, was measured for every test bar in every condition. Figure 10 shows that in conditions S, SE and SC2 (contaminated and not-optimally cleaned) the Failure Energy is not influenced by the clearance. However, in conditions Ref and SC1 (clean and optimally cleaned) there is a certain upward trend in Failure Energy with decreasing clearance (Figure 11) although the scatter is still large. Moreover, some rather high EtF values are measured for these conditions. It would appear that the Failure Energy method allows subtle influential factors to be detected and hence provides more results which are also quantitative.

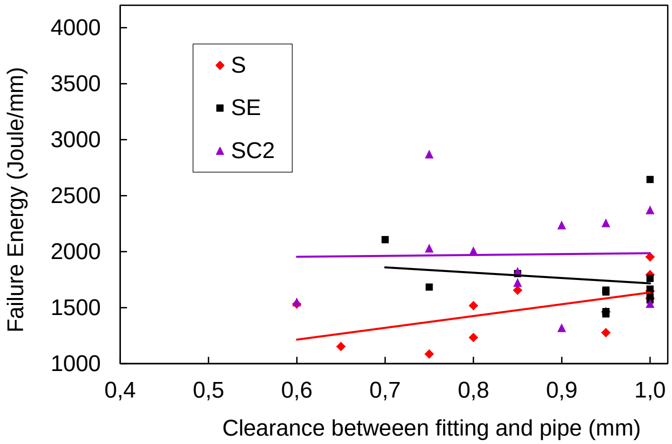


Figure 10. Failure Energy versus clearance between fitting and pipe as measured around the circumference of the joint in the conditions S, SE and SC2.

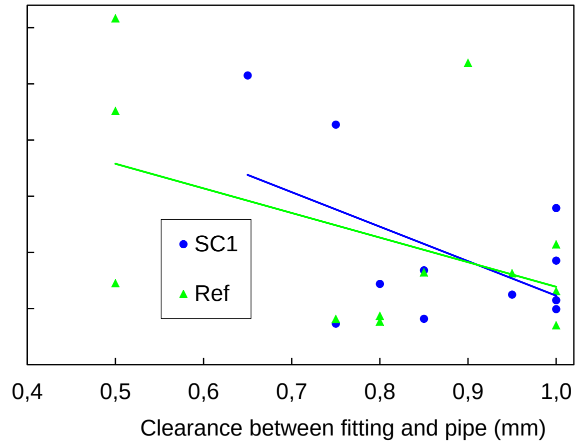


Figure 11. Failure Energy versus clearance between fitting and pipe in the conditions SC1 and Ref.

4. <u>*Isopropanol as cleaning agent*</u>

An unwanted phenomenon occurred during electro fusion work performed in the autumn (November) when constructing a new PE100 gas pipeline. A hissing sound was noted during fusion. The joints were cut out and inspected on site. Several unusual voids near the joint plane were observed with the naked eye (Figure 12). In the lab newly-cut samples were taken out of another part of the joint, which revealed more voids. A sample was cut from the wall of such a void. Two more samples were taken from the pipe and the fitting, in both cases far away from the voids. GC/MS analysis was performed only a few hours are cutting. The samples were heated at 210 °C during 15 minutes. Analysis of the vapours (Figure 13) revealed a large peak of a substance evaporated from the first sample that was absent in the latter two samples. MS identification proved that the large peak was due to the presence of isopropanol.

Figure 12. Voids caused by isopropanol evaporated during electro fusion. Voids sizes up to 2 mm.

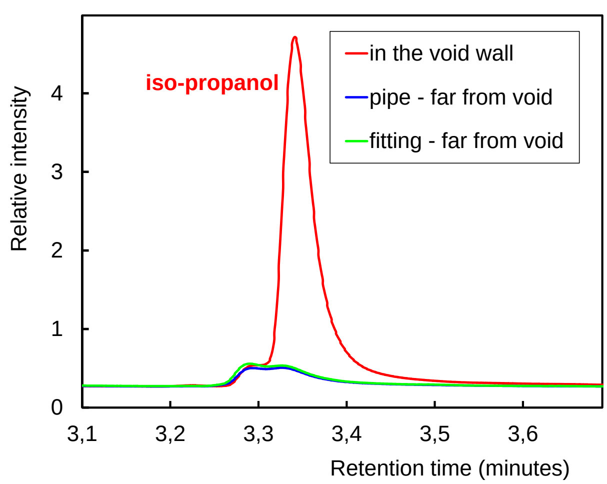


Figure 13. GC curve of three samples in an electro fused joint with vapour voids. Identification was made using MS.

Isopropanol had been recommended by the fitting manufacturer as a replacement for ethanol. However, no allowance had been made for the longer drying time needed for isopropanol in the given weather conditions (6 minutes instead of some seconds for ethanol). Because the short drying time intended for ethanol as prescribed in the jointing protocol was not amended, isopropanol did not have the time to evaporate completely. The high fusion temperature provoked evaporation of the isopropanol, which subsequently gathered to form vapour bubbles in the molten PE. These did not disappear during cooling of the joint. This led to the formation of the voids.

The use of isopropanol as a substitute for ethanol has two negative effects. The lower volatility of the former, as illustrated by the vapour pressure at 20 °C and the higher boiling point (Table 3) leads to a much slower evaporation in comparison to ethanol. Moreover, isopropanol dissolves somewhat better in PE (Table 3), as indicated by its Hildebrand Solubility Parameter [9]. This Solubility Parameter predicts how readily a liquid will dissolve in another substance, in this case PE. The closer the Solubility Parameter of a liquid is to that of PE (16.2 $MPa^{½}$), the more readily the liquid will dissolve in PE. Therefore, the fourth column in Table 3 predicts that isopropanol will dissolve more readily in PE than ethanol does. This effect on its own is also not beneficial to the evaporation rate of isopropanol.

Table 3. Boiling point and vapour pressure at 20 °C and the Hildebrand Solubility Parameter [9] of some relevant substances.

Substance	Vapour pressure at 20 °C (mbar)	Boiling point (°C)	Hildebrand Solubility Parameter ($MPa^{½}$)
Isopropanol	41.0	82	23.8
Ethanol	59.5	78	26.2
PE	-	-	16.2

DISCUSSION

Table 4 gives an overview of the contaminants identified in those tested joints from practice that had failed in a brittle manner in the Peel test. Three impurities were identified most often: silicones, silicates and calcium carbonate. These identifications are certain. Other identifications are only likely, such as human sweat, hand balms and a detergent. In some cases neither an identification nor a guess could be made. 39 Test bars have been investigated, but there were 41 observations, because in some cases two contaminants were present.

Table 4. Impurities present on 39 joint planes that failed brittle in the Peel test.

Contaminant	Certainty of identification	Number of observations	Percentage of cases (%)
Silicones	Certain	9	23
Silicates	Certain	18	46
Calcium carbonate	Certain	3	8
Sweat	Suspicion	2	5
Hand balms and a detergent	Suspicion	3	8
Unknown		6	15
Total		41	105

Although in 77 % of the cases (23 % + 46 % + 8 %) a definite identification could be made, it is not certain that the concentration of the identified substances was always high enough to explain brittle failure. Sometimes these concentrations were low, although detectable. It is not known what the threshold concentrations for these substances are in order to cause unacceptable deterioration of the joint quality. Relative concentrations can be measured from the infrared spectrum. This will be investigated in future.

That silicone contamination reduces jointing strength confirms the findings of Reynolds et al [6]. Various origins of silicones can be listed:

- Service engineers sometimes use silicone spray for lubrication of a variety of pipeline components.
- Certain hand balms or skin care products contain silicones as well.
- In the past electro fusion machines were sometimes serviced using silicone grease. After all these years remnants of such silicones may still be present on such fusion machines.
- The manufacturers of certain mechanical couplers use silicones to lubricate their own products without realising that this may cause cross-contamination of electro fused joints in the same pipeline construction project. Some manufacturers are now seeking to replace silicones.

From the reduced number of silicone contaminations in 2014 it would appear that the PE pipeline construction sector is becoming increasingly aware of the negative effect of silicone grease and oil on weld quality.

Silicates like clay dust also lead to a reduction in joint quality [4]. Marshall et al [3] found a reduction in the strength of electro fusion joints caused by talcum powder, which also is a type of silicate.
Silicates probably originate from fine particles that had been blown onto the pipe surface by the wind. The silicates are most likely soil particles (sand, clay).

In three cases calcium carbonate was present, which is known [4] to have a negative effect on joint strength as well. This points not to soil, but to fine cement particles. Cement consists of (among others) silicates and calcium oxide (“quick lime”, “burnt lime”). The latter particles, when very small, are readily converted to calcium carbonate particles in the presence of carbon dioxide in the air. The presence of calcium carbonate is a tell-tale sign that contamination from a nearby construction site at which dry cement is used has occurred.

There is one other important phenomenon involving the two types of inorganic particles (silicates and calcium carbonate). Inorganic particles are electrically non-conductive as is Polyethylene. Mechanical movement during rotation skinning of the pipe surface causes it to become electrostatically charged. This readily attracts fine particles, which then cling to the PE surface. The smaller such mineral particles are, the easier they are attracted by electrostatic forces, the more difficult they can be detected with the naked eye and the larger their negative influence on joint quality is [3]. Electrostatic charges may be removed using a liquid, but such a liquid may not readily remove tiny mineral particles.

CONCLUSIONS AND RECOMMENDATIONS

1. Contamination was found on the weld plane of electro fused joints that failed in a brittle manner in the Peel test. Silicones, silicate (sand and clay) particles and cement (calcium carbonate) particles were found most often (in 77 % of 39 cases).
2. In 13 % of cases the presence of sweat, hand balm and detergent was suspected. It is advised that welders should wear gloves of an appropriate type. In 15 % of cases the identification was inconclusive.
3. The investigation showed that assessing the Energy-to-Failure (Failure Energy) in the Peel test on test bars is an attractive and quantitative method and hence less subjective than visual examination.
4. There are differences in the ability of certain cleaning agents to dissolve silicone spray contamination from PE pipe surfaces. However, different liquids have different evaporation rates and a different solubility in PE. This must be taken into account when changing a cleaning liquid. Such a change inevitably leads to changes in the welding routine, which must be meticulously described in protocols used by welders in the field.
5. It is advised to take pipe ovality into consideration when testing electro fused joints.

ACKNOWLEDGEMENTS

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