Surface Characterization of Pipeline using Replica Techniques

SPEAKER
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Scheme of Presentation

- Introduction
- Research Question
- Objectives
- Experimental Methodology
- Results and Discussion
- Conclusion
Introduction

- The research is based on a Final Year Project conducted by students of DHA Suffa University, Pakistan. The project’s location is Shell Oil Kemari Terminal in Karachi.
- The project was aimed at validation and gauge efficacy of replica techniques to characterize surface defects on oil and gas pipelines installed at the project’s location.
Research Question

Surface Characterization of Pipeline Surfaces using Replica Techniques

Process and measure of material’s properties

A copy/reproduction of a material’s surface
Objectives

• To confirm the efficacy of Replica Techniques.
• To use Replica Technique as a tool to study failure characteristics.
• To present an alternate technique for in-situ failure analysis of a pipeline to Shell Pakistan.
• To evaluate the use of replica techniques in various on-field conditions. (The condition currently being investigated is of a pipeline that has been in-service for 20 years and is buried underground)
Experimental Methodology

Pipe Sections obtained from Shell Oil Kemari Terminal → CUTTING Sections cut to appropriate size samples → MOUNTING Samples mounted in a fast hardening resin → GRINDING Samples grinded on a surface grinder

POLISHING Samples polished → ETCHING → Metallographic Analysis of direct sample → AFM of direct samples surface

Replica of surface obtained → Metallographic Analysis of Replica Surface → AFM of replica sample’s surface → PROCESSING OF RAW DATA

FAILURE ANALYS based on data obtained → SERVICE LIFE PREDICTION
Testing Parameters

• **Sample Cutting (Power Saw)**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cutting Time</td>
<td>Till Sample Separates</td>
</tr>
<tr>
<td>Coolant</td>
<td>Machine Oil</td>
</tr>
<tr>
<td>Sample Obtained</td>
<td>Rings (25 mm depth)</td>
</tr>
</tbody>
</table>
Testing Parameters

- **Sample Cutting (Cut-off Wheel 54A25 – Al₂O₃ Resin)**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wheel Size</td>
<td>250 mm</td>
</tr>
<tr>
<td>Cutting Wheel RPM</td>
<td>3450 @ 60 Hz</td>
</tr>
<tr>
<td>Cutting Time</td>
<td>Till Sample Separates</td>
</tr>
<tr>
<td>Coolant</td>
<td>Tap-Water</td>
</tr>
<tr>
<td>Sample Obtained</td>
<td>Blocks</td>
</tr>
</tbody>
</table>
Testing Parameters

• **Sample Mounting (Resin: MultiFast-Black)**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure</td>
<td>100 bar</td>
</tr>
<tr>
<td>Temperature</td>
<td>Enough to melt the Resin</td>
</tr>
<tr>
<td>Soaking Time</td>
<td>5 mins</td>
</tr>
</tbody>
</table>
Testing Parameters

- **Surface Grinding (Grit Sizes 120-2000 successive increments)**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Revolutions per Minute (RPM)</td>
<td>200 Clockwise</td>
</tr>
<tr>
<td>Variable Normal Force</td>
<td>≤ 10 N</td>
</tr>
<tr>
<td>Specimen Mover RPM</td>
<td>200 Anticlockwise</td>
</tr>
<tr>
<td>Grinding Time Per Grit Size</td>
<td>≈ 60 Seconds ± 10%</td>
</tr>
</tbody>
</table>

Polishing of the samples was carried out after grinding, using 6 µ, 3 µ and 1 µ alumina suspension.
Testing Parameters

Etching (For Revealing Grain Structure)

• After polishing, samples were etched using Nital.
• Nital is a solution of alcohol and nitric acid commonly used for etching.
• The samples were dipped for about 10-20 secs in the etchant.
Metallographic Analysis

- Optical Microscopy is used to obtain micrograph at 100x magnification to reveal details for metallographic analysis in line with ASTM E112.
- The Average Grain Size calculation is then carried out.
- The micrograph reveals full and half grains.
Metallographic Analysis

Full Grain Boundary
Metallographic Analysis

- Magnification = print width / real width
- Number of grains = whole grains + 0.5 (partial grains)
- True area = (length x width) / (magnification)^2
- ASTM Grain size number $N = 2^{(n-1)}$
- $N = (\text{number of grains}) \times (0.0645/\text{true area})$
Metallographic Analysis

• From the equations, we find out the grain size to be 5.584
• The grain size falls into the ‘very fine’ grain size according to the ASTM reference table.
• The range of ‘very fine’ grain size being 5.6-11 microns.
• This gives us a measure that the sample has low ductility and increased hardness based on Hall-Petch Equation.
Replica Techniques

• RepliSet-T3 is used to generate our replica in CASE I.
• T3 is a two part silicon rubber containing a polymer and curing agent, a trademark product of Struers Denmark.
• It is applied on to etched samples to get results.
Optical Microscopy Results

Specimen

Replica
Optical Microscopy Results

Specimen

Replica
Optical Microscopy Results

Pitted Specimen

Replica
Optical Microscopy Results

- The accuracy of replica techniques can be observed from the Optical Results

Specimen  
Replica
Optical Microscopy Results

From the Results of Optical Microscopy, it can be concluded that:

• Replica technique was able to capture almost all salient features resolved at 100x on the sample surface and had almost the same feature size (X-Y) with very little shrinkage.

But is it capable to integrate the depth data?

Let’s use AFM!
Surface Characterization

- **ezAFM Scanning Parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scan Area</td>
<td>10 μm x 10 μm</td>
</tr>
<tr>
<td>Scan Speed</td>
<td>1 μm s(^{-1})</td>
</tr>
<tr>
<td>Scan Resolution</td>
<td>256 x 256 Pixels</td>
</tr>
<tr>
<td>Scanning Probe</td>
<td>PPP-CONTR (NANOSENSORS)</td>
</tr>
<tr>
<td>FT</td>
<td>1.98 V</td>
</tr>
<tr>
<td>Probe Normal Force Constant</td>
<td>0.2 N/m</td>
</tr>
<tr>
<td>Probe Diameter</td>
<td>9 nm</td>
</tr>
<tr>
<td>Probe Resonance Frequency</td>
<td>13 kHz</td>
</tr>
<tr>
<td>PID Values</td>
<td>15/1/17.8</td>
</tr>
<tr>
<td>(<em>Adjust as per requirement and cantilever’s oscillation</em>)</td>
<td></td>
</tr>
</tbody>
</table>

All samples were cleaned with \( \text{N}_2 \) before scanning with LFM.
Surface Characterization

• In order to answer our previous question, roughness of the samples was taken into account.
• The samples and their replicas were analyzed under AFM and their average roughness was measured.
Surface Characterization

- The graph shows a comparison of Roughness of the Specimen (Pink line) and its replica (Green line)
Surface Characterization Results

• The graphs shows the efficacy of Replica Techniques for Surface Characterization.
• The trend of the Roughness graph is seen to be similar for both the specimen and its replica.
• The range of Roughness is however, different.
Surface Characterization Results

• The range of Roughness for replicas was found to be 0 – 0.2 microns

• While the range of Roughness for the specimen was found to be 0.4 microns – 0.6 microns (considering smaller values to be error)

• From this, we can deduce to a certain extent that the Roughness of a sample reduces by a multiple of ‘2’ in its replica. However, more lab tests are being conducted by my team to affirm this claim.
Conclusions

• From the above discussion, we conclude.
  – As seen from Optical Microscopy, Replica is an effective tool to study Grain Structures.
  – The Grain Structure obtained is representative of characteristic material properties which can be evaluated using Hall-Petch Equation.
  – The properties can also help us determine the effective transition this surface will go through before its failure.
  – The details acquired by the replica matched with the original sample with very minimal error in 2D.
  – For 3D analysis, AFM was used, which helped us evaluate the average roughness in both original and replica surfaces. The roughness values follow a similar trend but do not present the same values.
Future Work

- More Replica Techniques will now be tested as per the guidelines made in this presentation in order to determine their suitability for various conditions.
- Depth is still required to be measured as a characteristic to ensure the lateral resolution of replica technique.
- Shrinkage of replicas under control temperature, humidity and pressure conditions is being carried out to evaluate its effective use for recording failure history.
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