

Preliminary Assessment of the Effects of Surface Preparation on IIT

Pooya Delshad¹, Peter Martin², Emily Brady³, Jeff Kornuta³, Jonathan Gibbs¹, Peter Veloo¹

¹Pacific Gas and Electric Company

²RSI Pipeline Solutions

³Exponent



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Abstract

Instrumented Indentation Testing (IIT) is a non-destructive technique increasingly employed to estimate the mechanical properties of pipeline steels, including yield strength and tensile strength. Accurate IIT results may depend on surface preparation, yet the specific effects of varying surface finishes on IIT outcomes remain underexplored. This study initially aimed to investigate the influence of surface preparation on IIT yield strength results. However, potential inconsistencies in the IIT results were observed to correlate with the presence of surface and subsurface manufacturing flaws. Metallographic cross-sections revealed defects such as rollovers and scabs, common in vintage pipeline materials due to historical manufacturing practices. These defects, penetrating up to 350 μm (~ 0.014 in.) below the original pipe surface, exceeded the depth of material removed during surface preparation for IIT (~ 250 μm / 0.010 in.). In defective material, IIT-determined yield strengths were decreased by as much as 10% relative to defect-free material. Our findings highlight the necessity of thorough surface preparation and defect detection methods to mitigate inaccuracies and ensure reliable IIT results.

Introduction

The nondestructive assessment of yield strength (YS) for in-field verification of line pipe materials is commonly achieved through surface indentation techniques like the instrumented indentation test (IIT). IIT is becoming increasingly recognized as an effective method for estimating the mechanical properties of pipeline steels without causing damage. As part of a Material Verification Program (MVP) [1,2,3], the authors have conducted extensive investigations to evaluate the quality and accuracy of IIT results. These investigations have primarily focused on validating IIT measurements against destructive tensile testing (TT) of various pipe features [4], developing in-house data processing to ensure transparency between physical measurements (load vs. depth) and the predicted yield strength (YS(IIT)) and tensile strength (TS(IIT)), analyzing experimental errors [5], and enhancing the accuracy of YS(IIT) by considering also the pipe chemistry [6]. During these activities, it was observed that while YS(IIT) is often within $\pm 10\%$ of the corresponding mechanical test value YS(TT), it occasionally deviates by 25% or more [7]. Figure 1 illustrates the behavior by comparing YS(IIT) versus YS(TT) for a set of 157 pipes.

To gain a deeper understanding of IIT performance, the authors examined various potential correlations between the accuracy of YS(IIT) and pipe characteristics typically assessed during material verification. These characteristics include seam type, chemical composition (e.g., %C, %Mn), grain size, the ratio of outer diameter (OD) to nominal wall thickness (NWT), and the magnitude of residual manufacturing stresses [8]. One potential contributing factor that remains underexplored in the industry is the effect of surface preparation at the IIT measurement site. PG&E's current procedure for surface preparation for IIT specifies that the minimum surface finish must be equivalent to or better than a 2000 grit abrasive (The IIT equipment manufacturer recommends 600 grit or higher, but a minimum of 400 grit). While the significance of surface

preparation for accurate IIT measurements is well acknowledged, systematic studies to examine the specific effects of different surface finishes on IIT results are scarce. While this work was initiated in the hopes of providing a conclusive report of the potential impacts of over- or under-preparation on the IIT results, thereby helping to explain some of the observed variability, unexpected findings emerged. Manufacturing defects, such as rollovers and scabs, that can be intrinsic to vintage pipeline materials were observed to be present even after the removal of 0.010" of material during surface preparation. These observations shifted the study's emphasis to explore the impact of these flaws, which were found to outweigh the effects of surface preparation alone.

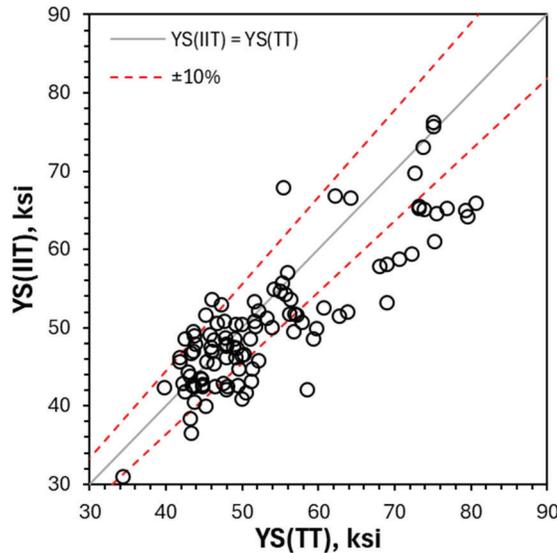


Figure 1. Comparison of yield strength determined from IIT versus tensile testing [8].

Materials and Methods

Pipe Samples

The pipe material used for this investigation came from a 20-in. OD, 0.52-in. WT, 1950s or 1960s pipe with a measured YS(TT) of 40 ksi and TS(TT) of 64 ksi.

Test Methods

Benchmark destructive tensile testing (TT) values for yield strength (YS(TT)) at 0.5% elongation under load (EUL) were obtained from uniaxial tension tests performed on full wall thickness 'straps' removed from each pipe. The straps were oriented in the transverse direction and were flattened in a press prior to testing. Nominal strap dimensions conformed to API 5L and ASTM A370, including gage length of 2.0 in. (51 mm), width of 1.5 in. (38 mm), and thickness equal to the full pipe wall thickness. Testing was performed in displacement control at a rate of 0.05 in./min (3×10^{-2} mm/s), and the reported values represent the average of four tests.

Yield strengths were also obtained nondestructively (NDT) by instrumented indentation testing, IIT, (YS(IIT)) using a Frontics AIS 2100 system equipped with a 500 μm diameter spherical indenter. The test sequence applied 15 displacement-controlled load-unload cycles, with the indentation depth increasing by 10 μm for each cycle to reach a final depth of 150 μm . The load-depth data were subsequently post-processed in R to convert the maximum load and the corresponding indenter depth for each cycle into representative stresses using the algorithm described in [8,9].

The surface of the pipe (at around 9:00 o'clock position) was prepared for IIT, using a sequenced process in accordance with the internal PG&E procedure. The surface was ground and buffed using the following grits in sequence: 40, 80, 280, 600, 1200, and 2000. The direction of buffing was alternated by 90 degrees between each grit. After each step, the surface was blown off with compressed air and cleaned with a rag and denatured alcohol to prevent scratching from remaining grit particles. For this study, the standard preparation was terminated at 3 intermediate levels (280 grit, 600 grit, and 1200 grit) and at the final standard level of 2000 grit (sample referred to as '2000' may also be referred to as 'Surface Study 2000' to differentiate from other 2000-grit locations).

A series of IIT measurements were collected in each preparation area and metallographic cross-sections were removed from the four preparation areas for inspection of the surface profile. Figure 2 shows a cutout from the pipe with the five preparation areas, and the included longitudinal (L) and transverse (T) metallographic sample locations, indicated.

In addition to the previously mentioned test locations (where standard preparation was terminated at three intermediate levels: 280, 600, and 1200 grits, and the final level of 2000 grit), the same pipe section underwent PG&E's standard IIT surface preparation procedure at the four clock positions to establish a baseline for the surface preparation study. Note that metallographic cross-sections were not prepared from these baseline test locations.

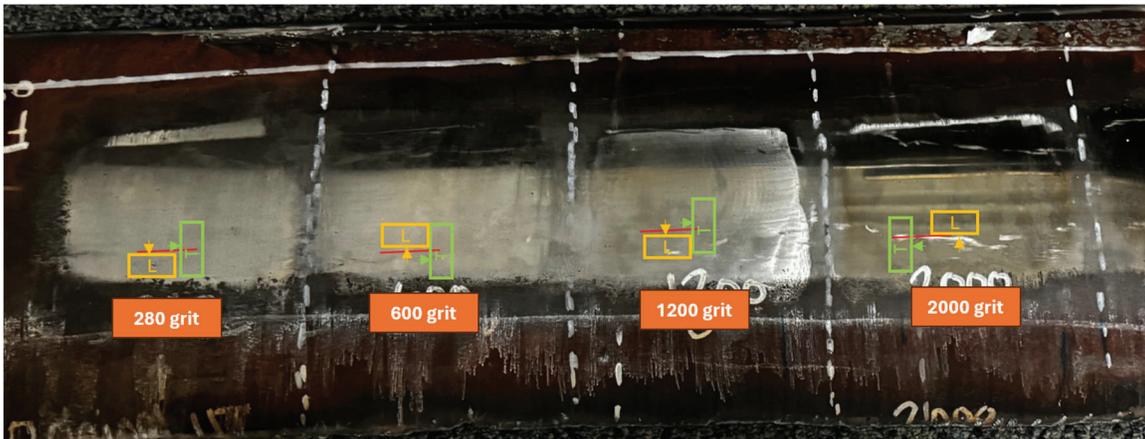


Figure 2. Cutout removed from the pipe section. The different surface finished are indicated by the grit: 280, 600, 1200, and 2000. The locations of the longitudinal and transverse metallography mounts are indicated by 'L' and 'T'. Red lines show the location of IIT indents.

Results

Figure 3a summarizes the IIT yield strength results for the four different surface preparation levels, 280 grit through 2000 grit. Each symbol represents the average of 9 or 10 IIT measurements at the designated location, the error bars represent the maximum and minimum measurements, the green dashed line represents the average yield strength from tensile testing on three transverse straps. The results suggest that testing after the coarse preparation (280 grit) results in the highest average yield strength, 44.0 ksi, with no overlap with the range of measurements from the other preparation conditions. The results from preparation at 600 and 1200 grit appear effectively equivalent, with respective averages of 38.9 and 40.2 ksi. Both averages are within the range of the measurement results from the opposing location. In contrast, the results from the location prepared to the standard level of 2000 grit suggest a lower average yield strength, 36.4 ksi. The maximum measurement at the 2000-grit location, 36.9 ksi, is below the minimum measurement from the other locations (600 grit), 37.8 ksi. From these results, one could erroneously conclude that the 2000-grit preparation may decrease the measured yield strength relative to 600 or 1200 grit. This will be explored in more detail below, where metallographic cross-sections from the four locations are presented which suggests it relates more to the presence of surface and sub-surface flaws.. For comparison, the average of three tensile tests performed on flattened transverse straps was 40.0 ksi, while the average of three tests performed on longitudinal straps was 35.8 ksi.

Figure 3b summarizes the IIT yield strength results from the four different areas prepared to 2000-grit final polish. The point for 'Surface Study 2000' duplicates the 2000-grit result from Figure 3a. The three additional points labelled 'Std' represent standard IIT performed at three clock positions, 3:00, 9:00, and 12:00, after standard surface preparation (i.e. ground and buffed using the following grits in sequence: 40, 80, 280, 600, 1200, and 2000). As stated above, the clock position for the surface study locations are adjacent to the 'Std 9:00' test location. The 2000-grit location from the surface study has a yield strength result of 36.4 ksi, while the standard test locations vary from 37.2 ksi at 3:00 to 40.9 ksi at 9:00.

A *t*-test comparing the results from 3:00 and 9:00 gives a *p*-value of 4×10^{-6} , Table 1, suggesting they are statistically different. In contrast, a *t*-test comparing the 9:00 and 12:00 locations yields a *p*-value of 0.03. This is below the 0.05 threshold value below which the likelihood that the two samples originated from the same population would typically be rejected (i.e. they are likely to be 'different'). Regardless, the *p*-value of 0.03 is still significantly higher than when comparing the 3:00 and 9:00 locations ($<1e-5$) or the 3:00 and 12:00 locations (0.002). The *p*-values in Table 1 suggest that the IIT results from the 'Std 3:00' location are statistically different from the other two 'Std' locations.

The raw load-depth data for the different test locations, Figure 4, suggests that the prior yield strength results are driven by changes in the indentation load during testing. In other words, the highest indentation loads are required for the 280-grit preparation and the lowest for the 2000-grit preparation, with 600 grit and 1200 grit clustered in the middle.

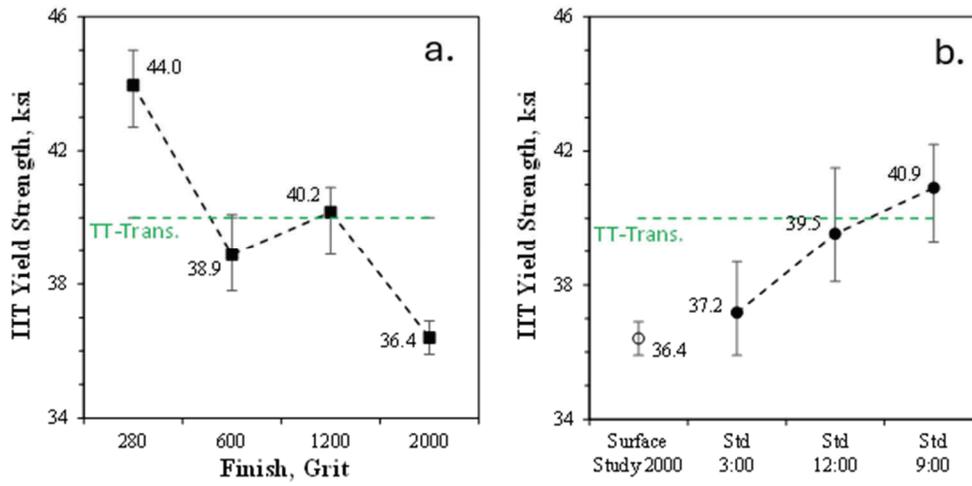


Figure 3. IIT yield strength (a) versus surface finish, and (b) for all locations with finish at 2000 grit. The error bars represent the maximum and minimum measured values.

Table 1. Results from t-test comparison of the 'Std' results from different clock positions

Loc. 1	Loc. 2	Mean 1, ksi	Mean 2, ksi	p-value
3:00	9:00	37.2	40.9	<10 ⁻⁵
3:00	12:00	37.2	39.5	0.002
9:00	12:00	40.9	39.5	0.031
Surface 2000	3:00	36.4	37.2	0.046

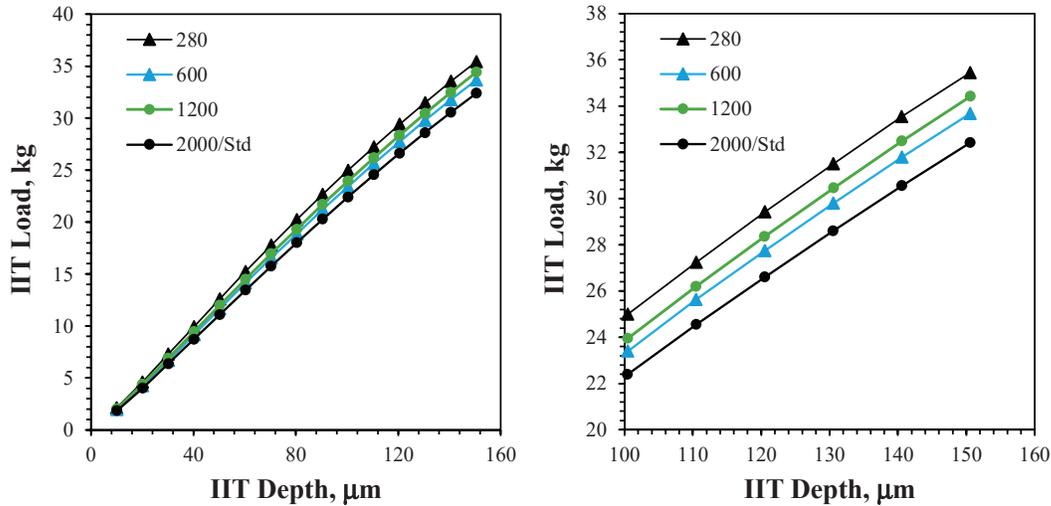


Figure 4. Load-depth curves for the different preparation conditions. The right-hand plot provides an expanded view of the upper part of the range shown at left.

Metallography:

Figure 5 shows the transverse metallurgical cross-sections of the OD pipe surfaces after polishing and etching with nital. These cross-section samples were taken from the areas with different preparation conditions, adjacent to the locations where IIT was performed. The images show typical grain structures for hot-rolled low carbon steels, comprised of relatively coarse grains of α -iron and well-defined colonies of pearlite. The orientation of the transverse cross-sections is indicated in Figure 5a, with the OD-surface at the top and the pipe circumference from left-to-right.

Figure 6 and

Figure 7 show the transverse and longitudinal metallurgical cross-sections, respectively, of the pipe surfaces (OD) at their as-polished condition. As can be seen, there are some surface/sub-surface features close to the outer diameter (OD) surface in all surface-prepared samples, indicated by red arrows. During the initial portion of the surface preparation process, approximately 0.010 inches of the surface was removed to ensure the decarburized layer on the OD surface was eliminated prior to the IIT. The surface/sub-surface features are clearly visible in the as-polished condition and appear as irregularly shaped, raised areas on the exposed surface of the pipe. In some samples, these features appear superficial (

Figure 6a), while in others, they penetrate deeper into the material (

Figure 6c). Upon closer examination of the microstructure in the areas with these features (

Figure 8), it appears that the microstructure at these locations is consistent with the microstructure in the rest of the sample.

This consistency suggests that the microstructure formed simultaneously with the rest of the material at high temperature (possibly during hot rolling process or heat treatment). There is no evidence of

plastic deformation in these areas, indicating that these features likely did not form as a result of the grinding or polishing process but originated from the manufacturing process. However, it is possible that these features were disrupted during the surface grinding process. It should be noted that in order to ensure the observed surface/sub-surface features are not artifacts of the cross-sections preparation and etching process, two sets of longitudinal and transvers cross-section samples were cut at two different areas (adjacent to the location of surface preparation area and IIT) and used for metallographic cross-section.

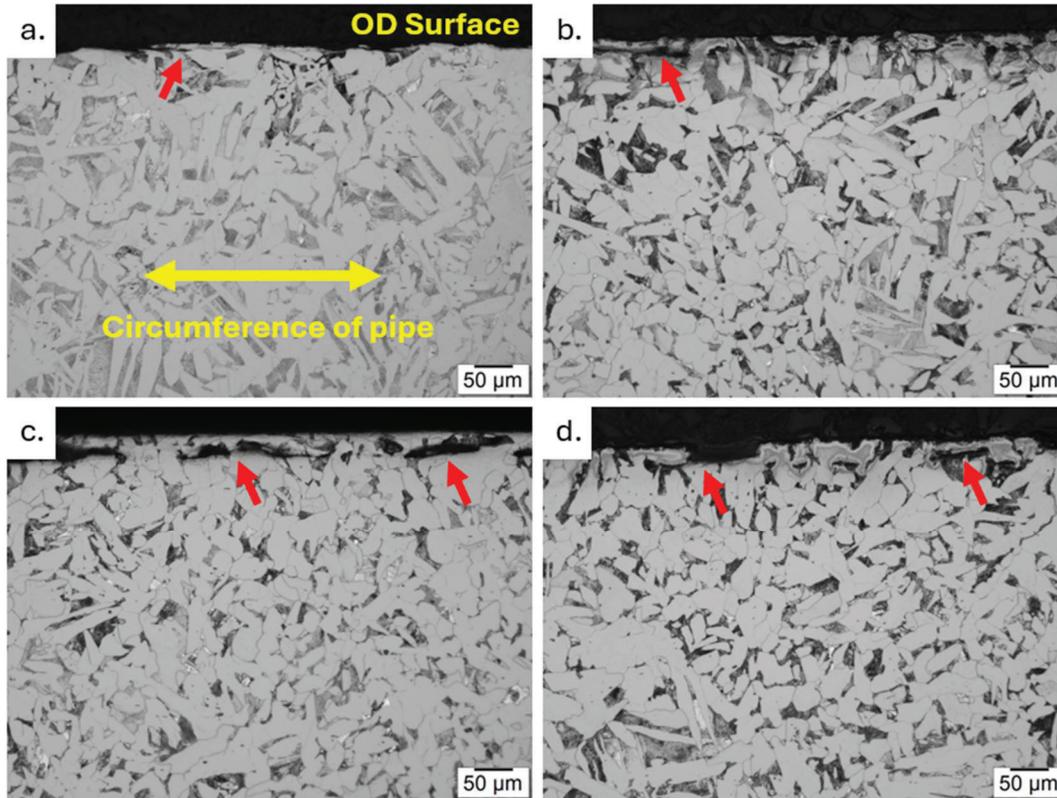


Figure 5. Transverse cross-sections of the surface after preparation (etched with Nital) to: a) 280 grit, b) 600 grit, c) 1200 grit, and d) 2000 grit.

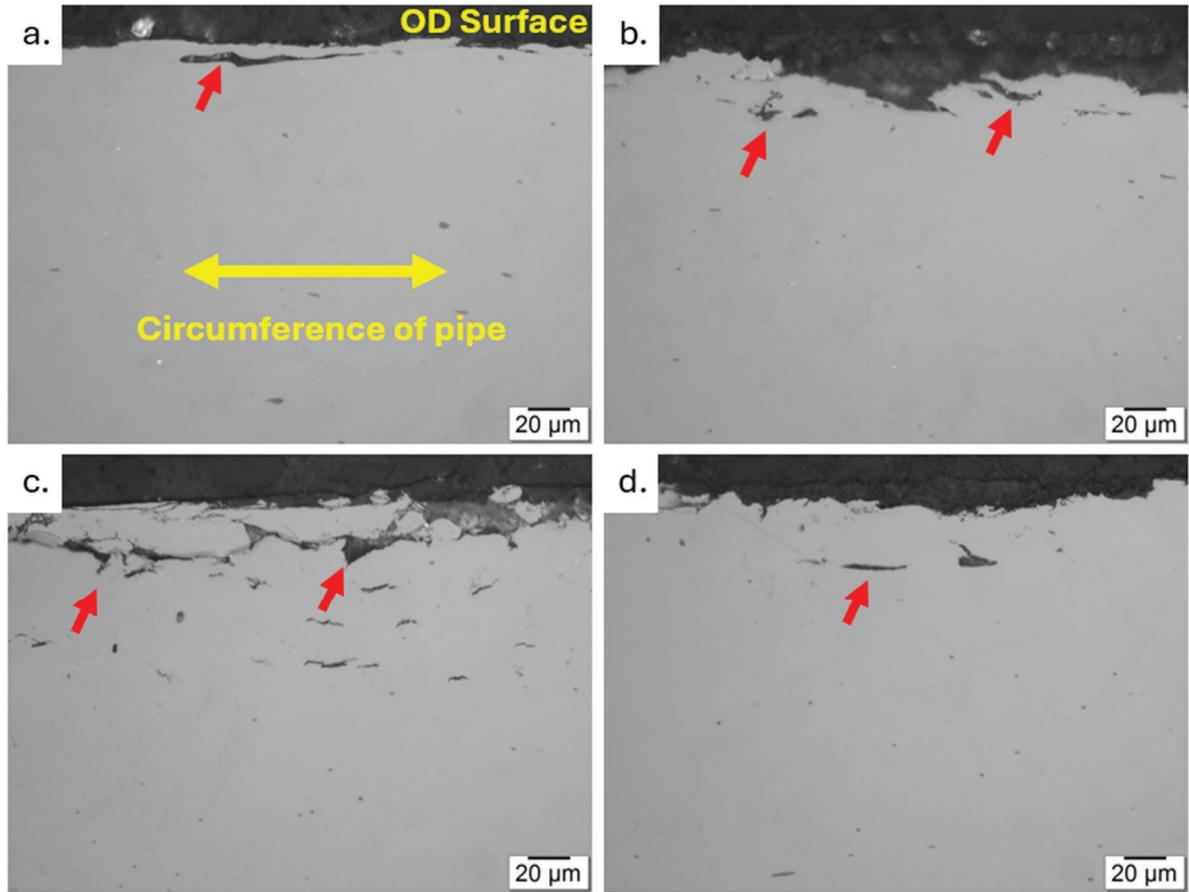


Figure 6. Transverse cross-sections of the surface after preparation (as-polished) to: a) 280 grit, b) 600 grit, c) 1200 grit, and d) 2000 grit.

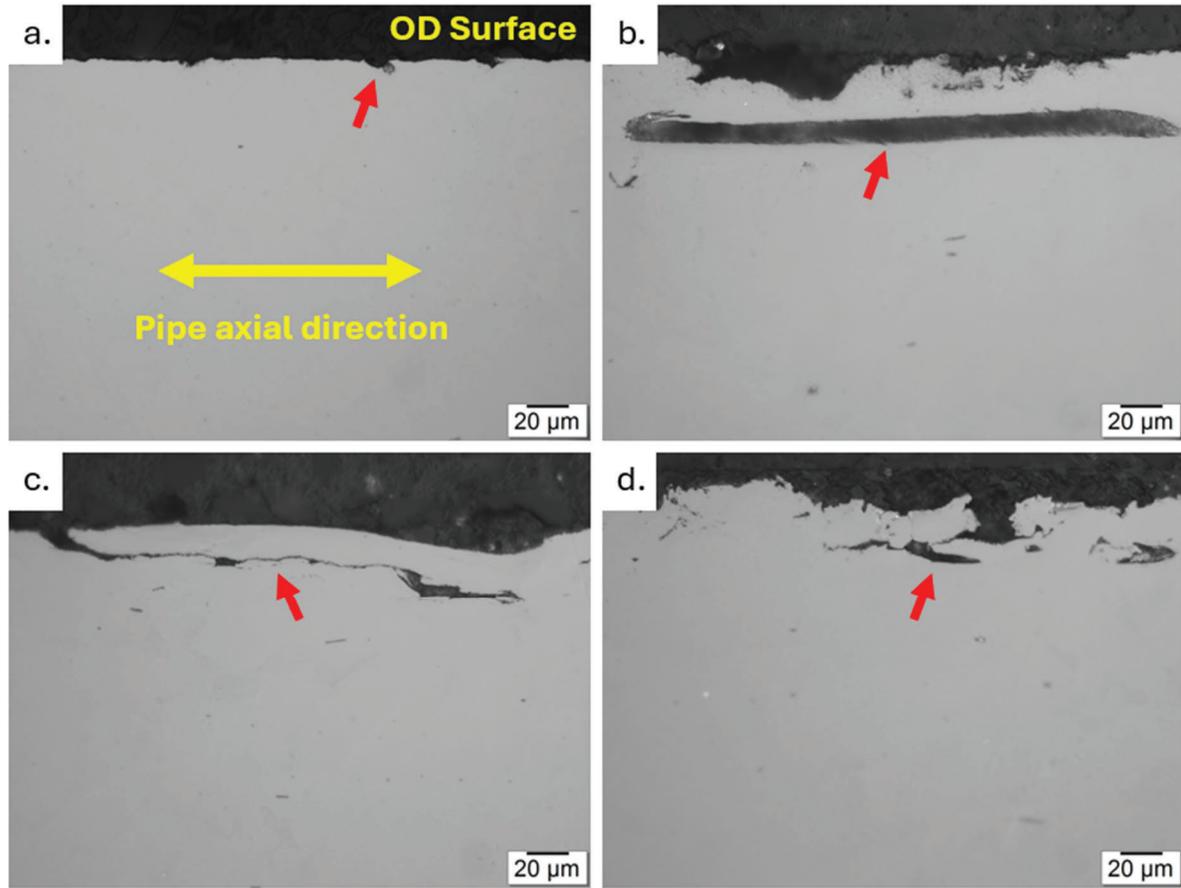


Figure 7. Longitudinal cross-sections of the surface after preparation (as-polished) to: a) 280 grit, b) 600 grit, c) 1200 grit, and d) 2000 grit.

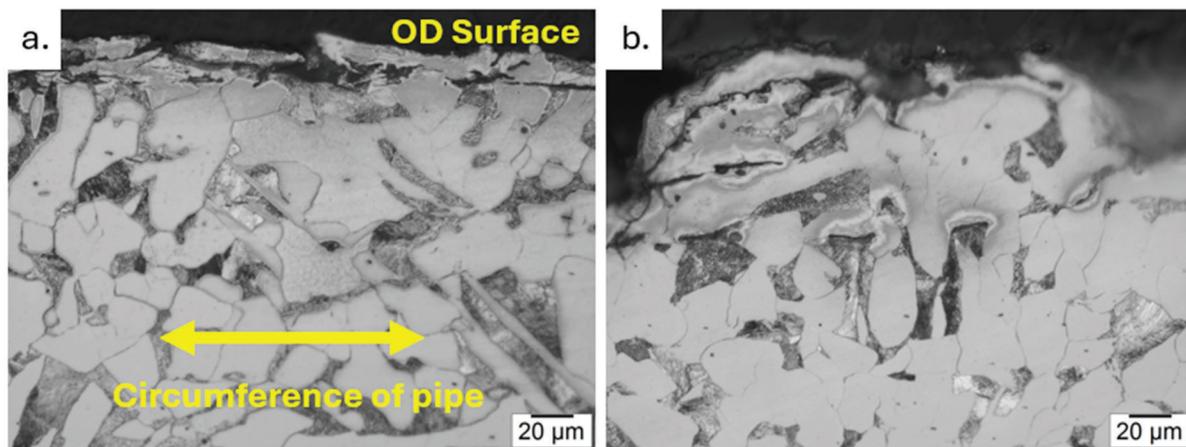


Figure 8. Transverse sections prepared to: a) 600 grit and b) 1200 grit.

Discussion

In this study, the influence of surface preparation on the results of instrumented indentation testing (IIT) was investigated in an attempt to understand potential variability between yield strength values obtained through IIT and those determined by destructive tensile testing. During the examination of metallographic cross-sections from locations subjected to IIT with varying surface preparation conditions, we observed surface and sub-surface features indicative of manufacturing flaws. These flaws likely formed during high-temperature processes, such as hot rolling or heat treatment, as the microstructure of these regions was consistent with the base material of the pipe. However, our analysis reveals that the presence of these features complicates the interpretation of surface preparation effects on IIT results.

Manufacturing flaws such as scabs, rollovers, and voids are intrinsic to pipe production processes [10] and are particularly prevalent in vintage pipelines due to the limitations of historical manufacturing techniques and quality control practices [11]. Two examples of pipe surface manufacturing defects are shown in Figure 9. These known manufacturing flaws exhibit similarities to the features observed in the current study (e.g., Figure 7c) and demonstrate that the defects can occur with depths exceeding the material removal during surface preparation for IIT. These defects can introduce non-uniformities that disrupt data acquisition during IIT, resulting in increased data scatter and potential misinterpretation of material properties. Specifically, such defects can affect the repeatability and reproducibility of the tests, introducing challenges in statistical analyses.

Observations from the metallographic cross-sections in this study (Figure 6 and Figure 7) suggest that these flaws may encapsulate voids or impurities, which exhibit elastic and plastic properties distinct from those of the surrounding base metal. Furthermore, random surface roughness caused by these defects can interfere with the proper seating of the testing equipment, thereby impacting force distribution and indentation accuracy.

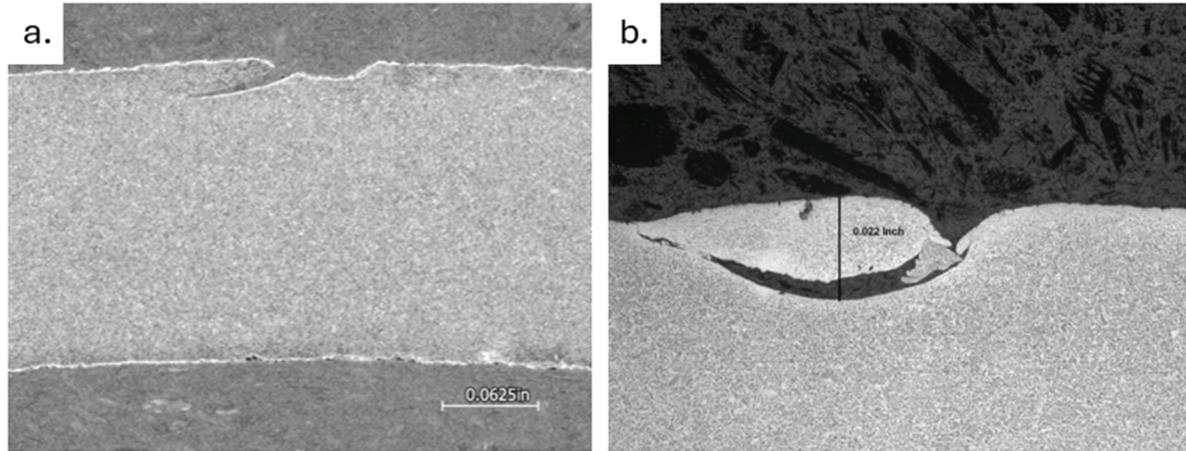


Figure 9. Example of pipe manufacturing surface defects [10].

Given these challenges, surface preparation plays a critical role in ensuring reliable IIT results. Grinding and polishing are essential for removing surface defects and achieving a smooth, uniform testing surface. Pre-test non-destructive evaluation (NDE), such as magnetic particle inspection (MPI), is recommended to detect and map surface and subsurface defects before conducting IIT. However, our findings suggest that MPI performed pre-polishing may not identify all subsurface features, as additional defects may be exposed during grinding and polishing. Therefore, performing MPI after surface preparation and prior to IIT may be appropriate for the detection of underlying defects.

This ongoing study highlights the significant impact of manufacturing flaws on IIT results and underscores the need for a systematic approach to surface preparation and defect detection. Future research will aim to conduct controlled experiments to isolate the effects of surface preparation without the presence of manufacturing defects. These efforts will provide deeper insights into optimizing IIT procedures for more accurate yield strength assessments in pipeline materials, particularly those from vintage pipelines where manufacturing flaws are more prevalent.

Summary and Conclusions

This study initially set out to investigate the effects of surface preparation on IIT yield strength results and to address the variability observed compared to tensile testing. However, the work ultimately revealed that manufacturing flaws play a far more significant role in influencing IIT outcomes than differences in surface preparation. This unexpected finding shifted the focus of the study and underscored several critical insights:

- IIT Yield strength results can vary by 10% between locations prepared by nominally the same methods. This variability, historically attributed to material heterogeneity, may also arise from preparation-induced subsurface damage.
- The presence of manufacturing defects such as rollovers, scabs, and voids affects the reliability of IIT results, introducing variability and inconsistencies in the data. Metallographic examinations revealed that these features, inherent to the manufacturing

- process, persisted despite rigorous surface preparation and could alter IIT outcomes, potentially overshadowing the influence of surface preparation.
- Comparable IIT yield strength values were obtained from surfaces prepared to 600, 1200, or 2000 grit, indicating that a range of intermediate finishes may suffice for reliable measurements. However, further testing across multiple locations is required to confirm this equivalence.
 - This study suggests that it might be appropriate to perform magnetic particle inspection (MPI) after surface preparation for IIT to determine the presence of pre-existing manufacturing flaws that could have been exposed by material removal during surface preparation.
 - These findings highlight the need for a systematic approach to both surface preparation and defect detection to ensure accurate and reliable IIT assessments for pipeline materials.

Future work suggestions

- Repeat the experiment on similar grade (e.g., hot-rolled grade B) and higher grade pipes (e.g., X52 and X65), and perform IIT on areas free from surface and subsurface flaws under different surface preparation conditions.
- Investigate appropriate NDT methods, such as MPI, to detect surface-breaking flaws after surface preparation.

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