

# NIST Interagency Report NIST IR 8535

# Mechanical Metallurgy on Columbia Gas X100 Experimental Pipe

Timothy "Dash" Weeks Ryan M. White Jake T. Benzing Enrico Lucon Nicholas Derimow Ashley Kroon Robert Smith

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# Mechanical Metallurgy on Columbia Gas X100 Experimental Pipe

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### Abstract

This study evaluates the material properties of an X100 pipeline steel extracted from an experimental transmission pipeline section placed into service in the 1960s. The purpose is to compare these properties with current X100 steel standards. Comprehensive chemical characterization, microstructure analysis, and a series of mechanical tests—including tensile, Charpy impact, and indentation tests—were conducted to assess the long-term stability and reliability of these early high-strength steels. The analysis involved seven different welded sections of the pipeline to account for potential variations within the material and welding procedures.

The findings indicate significant differences in chemical composition across the various pipe sections, suggesting that these sections may represent different experimental materials. The base metal predominantly exhibited a bainite-ferrite microstructure, with noticeable variations near the pipe surfaces. Unannealed girth welds demonstrated higher toughness and increased hardness in their heat-affected zones compared to seam welds. While most sections of the vintage X100 steel met the modern tensile property requirements, several sections did not meet the impact toughness criteria.

In conclusion, the experimental X100 steel aligns with current tensile property requirements, however the steel fails to meet current toughness related requirements. The observed differences in testing methods between current standards and those published in the 1960s were minor, and therefore not a convincing source for observed property differences considering all sources of uncertainty. The absence of original pre-service mechanical testing data prevents conclusions about time-dependent property changes.

#### Keywords

Pipeline steels; X100 pipe; Welding; Metallurgy; Mechanical engineering.

#### **Executive Summary**

This report provides a comprehensive analysis of the vintage Columbia Gas X100 pipeline steel that was pulled from service. The analysis includes chemical composition, microstructure, and mechanical properties. The physical metallurgical analysis was conducted using various techniques such as optical emission spectroscopy, optical microscopy, scanning electron microscopy (SEM), and electron backscatter diffraction (EBSD), whereas the mechanical metallurgical analysis was conducted using notched impact testing (Charpy V-Notch, CVN) and tensile testing.

The primary objectives of this study were to determine if age dependent property changes could be determined. Another key objective was to determine if the extracted line pipe would meet current standards. The report is divided into several key sections, each focusing on different aspects of the analysis.

Chemical Analysis: The results indicated inconsistencies in chemical composition across the seven pipe sections, suggesting that each section might be a different experimental material. Despite these inconsistencies, the pipe sections displayed a bainite-ferrite microstructure typical of X100 pipeline steel.

Microstructure Analysis: The microstructures of the base metal, heat-affected zones (HAZ), and welds were examined using optical and SEM imaging, as well as EBSD. The analysis revealed significant variations in grain size and misorientation within the HAZ. The findings showed that the welds and HAZ contained a mix of martensite, ferrite, and bainite, consistent with previous studies on X100 pipeline steel.

Tensile Testing: Tensile tests were performed on full-thickness flattened strap specimens from the pipe body and seam welds. The results showed that most pipe sections met the current API 5L X100Q requirements, with few exceptions that are mostly due to small sampling sizes. Differences in testing methods between the time of production and the present day were noted, particularly regarding the accuracy of measurement instruments and data acquisition systems.

Instrumented Charpy Testing: Charpy impact tests were conducted on third-size specimens from the base metals, weld metals, and HAZ. The results indicated that the base metals in the longitudinal orientation exhibited high upper shelf energies (USE) and low ductile-to-brittle transition temperatures (DBTT). However, most specimens tested in the transverse orientation did not meet the API 5L requirements. The seam weld metals and HAZ also showed varying degrees of toughness, with only a few specimens meeting the required standards.

Indentation Testing: Vickers and Knoop hardness tests were performed on the base metals, seam welds, and girth welds. The hardness profiles showed an increase in hardness in the HAZ adjacent to the weld cap, while the base metal and weld metal had similar hardness levels. Nano-indentation tests revealed a minor decrease in hardness near the internal pipe surface but no significant change in elastic modulus.

Discussion and Comparison: The report discusses the implications of the findings, highlighting

the challenges in comparing vintage and modern pipeline steel due to differences in testing methods and standards. The vintage X100 steel line pipe was found to be comparable to modern X100 steel in terms of tensile properties and performance. However, the variations in chemical composition and microstructure as well as low and varying toughness results underscore the need for careful evaluation of vintage pipeline materials.

In summary, this report provides valuable insights into the properties and performance of a Columbia Gas X100 pipeline steel, offering a foundation for future research and assessment of similar materials in the pipeline industry. This report further underscores the importance of having access to complete material data at the time of manufacture to accurately detect age dependent property change, especially relevant to degradation, resulting in increased risk and decreased system reliability.

This report is the final deliverable to meet the requirements of the interagency agreement (IAA) between NIST and the U.S. Department of Transportation – Pipeline and Hazardous Materials Safety Administration (DOT/PHMSA). The IAA was funded under DOT contract 693JK319N000013-001, more details about this research project can be found on the DOT/PHMSA website under project number 863. https://primis.phmsa.dot.gov/matrix/PrjHome.rdm?prj=863

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### 1. Introduction

#### 1.1. Motivation

The US Department of Transportation (DOT), Pipeline and Hazardous Materials Safety Administration (PHMSA), in coordination with the Department of Commerce, National Institute of Standards and Technology (NIST), are congressionally mandated to carry out a program of research, development, demonstration, and standardization to ensure the integrity of pipeline facilities. This requirement was instituted via the Pipeline Safety Improvement Act of 2002 and subsequently amended by the Pipeline Safety, Regulatory Certainty and Job Creation Act of 2011; and 15 U.S.C. §§ 272(b)(5), (10), (11) and 272(c) authorizing NIST statutory authority to undertake these activities. These Acts highlight that NIST shall evince its expertise in materials research and assist in the development of consensus technical standards, as that term is used in section 12(d)(4) of Public Law 104–13 (15 U.S.C. 272 note) and provide an opportunity for PHMSA to seek material research expert services from NIST.

The research herein includes mechanical metallurgy on X100 pipeline steel extracted from an experimental transmission pipeline section placed into service by Columbia Gas in the early 1960s. DOT/PHMSA has expressed an interest in detailed studies on the mechanical properties of the pipe via microstructural analysis, tensile testing, Charpy testing, and hardness mapping. These studies have been conducted on both this pipe prior to use and on a more modern X100 (circa 2000s) and will therefore facilitate discussion about property changes due to time- and service-related degradation and property differences due to chemistry and microstructure. This work extends these studies with the addition of instrumented sub-size Charpy testing, and instrumented nano-indentation. The mechanical properties are correlated with microstructure and chemical information via analytical electron microscopy, thereby providing processing-structure-property-performance relations for the pipe provided and a more modern X100. NIST participated in and conducted research on a modern X100 pipeline steel and girth welds under DOT/PHMSA Research Project DTPH56-07-T-000005, Weld Design, Testing, and Assessment Procedures for High Strength Pipelines [33], Weld Design, Testing, and Assessment Procedures

for High Strength Pipelines [1]. In all studies, base metal, heat-affected zones (HAZs), and weld metals were examined.

#### 1.2. Background

The American Petroleum Institute (API) Specification for Line Pipe (5L [6]) [2] is such that the label, X100, for example, refers to line pipe with 100,100 psi (690 MPa) minimum yield strength. The Columbia Gas System Service Corporation [3] demonstrated the feasibility of utilizing a higher strength X-series steel with minimal economic drawbacks. Such steel was suggested for implementation into onshore gas transmission pipelines [4]. A comprehensive review on the welding of oil and gas pipeline steels suggested a shift from line pipe grades X65, X70, and X80 to higher strength such higher strength grades such as X90, X100, and X120 [5]. These low alloy, carbon steels had previously exhibited fully ductile fracture behavior during full-scale burst tests at ambient temperature [6] [7].

API 5L [2] will be used here as the reference requirements for the pipe body properties. While some tests reported here include seam weld properties, there are no weld procedures (from the 1960's) or weld qualification requirements (from the 1960's) available to compare the provided X100 joints and seams. Moreover, 21<sup>st</sup> century weld procedures and qualification requirements are unlikely to be applicable to the vintage materials. Additional weld material has been retained for potential future work in this area of interest, but no previous weld data is available for a suitable time-history comparison. Charpy V-Notch (CVN) testing was performed on specimens notched to characterize base metal (BM), weld metal centerline (WMC) and heat affected zone (HAZ) material as part of the requirement for API 5L.

A European Commission report on the mechanical characteristics of API 5L - X100 quantified the mechanical properties and fracture resistance in both plate and pipe for evaluating the effects of pipe forming, as well as defect damage tolerance requirements, and correlated ductile-to-brittle transition temperatures to previously measured values [8]. It has been shown that there existed anisotropic behavior in X100 plates between the rolling and transverse directions [9], and was investigated by the Materials Reliability Division of NIST in 2008 [10]. Pre-strain from forming reduces ductility and crack growth resistance [11]. Environmental factors have also been investigated in X100 steels with respect to strain-aging [12], [13], [14], hydrogen embrittlement

[15], [16], [17], [18], carbonate corrosion [19], simulated bitumen [20], simulated soil [21], oilfield produced water and brines [20], H<sub>2</sub>S [23], and NaCl with Mg and Ca [24]. The ductile fracture behavior of X100 has also been characterized in previous studies [25], [26].

The microstructure of X100 has been described as ferritic/bainitic, containing martensiteaustenite (MA) [15], as well as acicular ferrite [27]. The effect of the crystallographic texture on the mechanical properties of X100 has also been investigated with respect to finish rolling temperature [28], [29]. The strengthening mechanisms have been attributed to solid solution strengthening, as well as grain boundary, dislocation, and precipitation strengthening [30]. The relationship between crystallographic texture (from rolling) and fracture behavior have also been studied via Charpy impact toughness on X100, where it was observed that controlled rolling processes can result in superior impact toughness when performed at lower temperatures [31].

A series of studies in 2011 reported on the development of optimized welding of X100 [32] and mechanical testing and assessment of X100 welds [1]. They were both prepared for the Design, materials, and Construction Technical Committee of Pipeline Research Council International (PRCI), and the United States Department of Transportation Pipeline and Hazardous Materials Safety Administration (PHMSA). Later studies investigated the on-scene weldability of X100 pipelines [33], microcracks of X100 weld joints heat affected zones (HAZs) [34], as well as structure-property-fracture mechanisms [35]. The morphology of the bainite in the nugget zone in X100 welds was found to be affected by the stirring tool material during friction stir welding [36].

#### 1.3. Historical Pipe and Data

In 1964, the Columbia Gas System Service Corporation (Columbia Gas) requested the United States Steel Corporation to investigate the possibility of producing a large-diameter pipe with a minimum yield stress of 100,000 psi (689 MPa). Columbia Gas produced and installed a section of this pipe parallel to an existing 26 in (0.66 m) pipe. The test section consisted of 1,193 ft (366 m) of 36-inch (0.91 m) OD X100 pipe with a wall thickness of 0.25 inches (6.35 mm) and short sections of X60 with a wall thickness of 0.45 in (11.4 mm). Columbia Gas capped both ends of the 1,193 ft (366 m) section of pipe and connected it to the main transmission pipeline

A free copy of this report can be obtained from: https://doi.org/10.6028/NIST.IR.8535 via a two-inch (50.8 mm) line, making the experimental pipe serve simply as a pressure vessel. The experimental pipe did not transmit gas.

R. S. Ryan described the pipe and installation process in detail [1], though much of the publication focused on the nuances of installing the pipe, including grading/ditching, coating, backfill, and bending long (20 ft - 60 ft/6 m - 18 m) sections of pipe. In the paper, R.S. Ryan noted that US Steel formed the pipe into cylinders, welded it along the seam, and then quenched and tempered it. US Steel heat-treated the seam welds producing the pipe section before field deployment. Workers connected separate sections of 20-foot (6 m) pipe via automated or semi-automated girth welds.

The mechanical property data provided with the original pipe is sparse. The "minimum yield strength" is given as 100,000 psi (689 MPa) with a minimum tensile strength of 115,000 psi (793 MPa) and an elongation of 15 %. There is also a singular Knoop indentation trace across a girth weld (shown in Figure 1) and two graphs related to Charpy impact testing: impact energy and shear area (shown in Figure 2).



Figure 1 - Knoop indentation trace across a girth weld, from R. S. Ryan (1965).



Figure 2 - Charpy test data (absorbed energy on the left and shear fracture on the right) reported by R. S. Ryan (1965).

Unfortunately, these are all the historical data that were provided about the pipe. In the paper from 1965, R.S. Ryan makes three references to other sources which may provide information about the historic X100 pipeline steel:

- "A further indication is the performance of a similar material in the Athens tests."
- "Generally, the shipping instructions were based on the work done by the AGA NG-18 program."
- "There are several ways to increase yield strength beyond the present levels of the semikilled steels, *i.e.*, fully killed steel, alloy steel, and heat treatment such as normalizing or quenching and tempering."

The first quote references the Athens tests, which were a series of full-scale rupture tests carried out by the Battelle Memorial Institute in Athens, Ohio [37]. The second quote references the American Gas Association NG-18 program, which was a program conducted over approximately 40 years in the mid to late 1990's. The comprehensive NG-18 program investigated several steel chemistries and testing geometries to produce crack models in natural gas pipeline steels and develop strategies to arrest existing cracks in pipelines [38]. The historical work that was referenced by R.S. Ryan most likely refers to a 1963 NG-18 report [39].

The third quote indicates that the experimental X100 pipe is of the semi-killed type, where the steel is partially de-oxidized during production, typically using silicon. The reduction in oxygen content in semi-killed steel reduces the development of porosity (via the production of CO bubbles) during welding, therefore increasing weldability. In the case of a fully killed steel,

aluminum is used in place of silicon to further reduce the oxygen content and improve weldability. The reference to semi-killed steel in the R. S. Ryan manuscript may indicate some expectation of the chemistry of the experimental X100 pipeline steel. A 2005 report on vintage pipelines authored by Battelle Memorial Institute discusses these various processes with respect to historical timeline and implementation [40].

The Charpy tests performed in this study were conducted on third-size specimens (thickness B = 3.3 mm, width W = 10 mm). This geometry was chosen for the following reasons.

- The reported pipe thickness was 0.25" (6.35 mm) and therefore insufficient to extract full-size Charpy specimens. This necessitates the use of subsize specimens since miniaturized samples had not yet been introduced in the 1960s.
- The energy levels reported in the R. S. Ryan manuscript are indicative of a sub-size geometry and the third-size specimen dimensions result in the maximum material use given the wall thickness limitations.

The orientation of the samples (longitudinal or transversal) is also undocumented in the R. S. Ryan manuscript, while it is reasonable to assume the data in Figure 2 were obtained from the base metal (pipe body).

Open literature resources were exhausted with remaining unknowns about the provided X100 steel. PRCI provided 37 documents related to the AGA NG-18 program. Steels described in these reports were compared to the current knowledge of the vintage X100 pipeline steel and will be discussed where appropriate, yet specific details remain copy protected by PRCI.

Additional information about the historic pipe was sought from TC Energy (previously Columbia Gas). TC Energy was able to provide some historical documentation, but mainly consisting of:

- Records of hydrostatic tests of installed pipe sections.
- Visitor lists and lists of "Workers on Atlantic Seaboard Corporation's 36" X-100 Experimental Line".
- A failure report for a seam weld that failed in the field during installation.
- Invoices and receipts for the use of an AMF welder and miscellaneous supplies for use during the installation of the pipe section(s).

The historic documentation provided by TC Energy is included in Appendix A. One especially notable document is a letter describing the hydrostatic testing of the "36" Line WB-5." In this letter it is noted that:

- The X-60, .438 Wall Pipe in Sections 1, 2, 4, 5, and 6 has a specified minimum yield of 1460 PSI.
- The original test sheets will remain on file at Dranesville.

This indicates that the thicker pipe wall sections, P3S1 and P3S2, are likely X60 grade material (from 1967), which is not of specific interest to this project. Mechanical testing will be carried out on the base metal, but the multi-thickness, multi-material welds do not provide any insight into the welding of the vintage X100 material.

The second note that the original test sheets will remain on file at Dranesville indicates that there was at one time a stock of original material. Despite communication with US Steel, TC Energy (formerly Columbia Gas), and PRCI, no original material could be located.

## **1.4. Project Output**

There is little data available that is verifiably associated with the X100 pipeline steel referenced in the R.S. Ryan paper. Furthermore, there is no data available that is verifiably associated with the exact sections tested at NIST. Without control specimens (*i.e.*, original X100 pipeline that was not buried or pressurized with natural gas) or data from the original pipe, there is no appropriate way to verify any change or degradation in microstructure, chemistry, or mechanical properties of the line pipe steel provided to NIST. The only data that can be referenced is the minimal Charpy impact and hardness indentation data provided by R.S. Ryan.

As an output to this project:

- The collected data will be compared to the original indentation presented by R.S. Ryan whenever possible.
- All collected data (raw and processed) will be provided in a repository as baseline data for future researchers.
- All specimens and material from the project will be provided to the Pipeline Research Council International (PRCI) for inclusion in their warehouse of pipeline materials for use by future researchers.

The data repository for this project can be found at the following location:

https://doi.org/10.18434/mds2-3322

# 2. Materials and Methods

# 2.1. As-Received Materials

Three pipe sections of the experimental X100 pipeline were received at NIST and the cut plan that was devised based on the test requirements prescribed by API 5L. The three pipe sections, named Pipe 1 (P1), Pipe 2 (P2) and Pipe 3 (P3), all contain a variety of welds, and pipe body sections with different wall thicknesses, and welding methods. Each pipe body section was formed from quenched and tempered plate, therefore giving them a designation of X100Q, and being categorized as a PSL 2 pipe with submerged arc welded longitudinal seams (SAWL/LSAW). These designations are based on current API 5L line pipe specifications since X100 (Q or M) was not included in the specifications at the time of manufacture. The pipe section dimensions, and construction details are given in Table 1.

The pipe construction details and measurements were collected, and three-dimensional (3D) computer aided design (CAD) software was used to create digital models of the pipes. These CAD models were used for subsequent sectioning plans to ensure that specimens were extracted from known locations in the pipes with respect to key weld features in the pipes. Pipe details can be found in Appendix B.

Pipe Section	Designation	Total Length	Internal Diameter	Wall Thickness	Seam Welds	Girth Welds	Seam Offset
Pipe 1	P1S1	49.5 in (1.26 m)	35.1 in (89 cm)	0.26 in (6.6 mm)	1	1	154°
(P1)	P1S2				1		
Pipe 2	P2S1	47.25 in (1.2 m)	35.1 in (89 cm)	0.26 in (6.6 mm)	1	1	20°
(FZ)	P2S2				1		
	P3S1	97 in (2.46 m)	35.1 in (89 cm)	0.45 in (11.4 mm)	1	2	95° (S1 to S2)
Pipe 3 (P3)	P3S2			0.45 in (11.4 mm)	1		26° (S1 to S3)
	P3S3			0.26 in (6.6 mm)	1		
Section Designations include the pipe number and the seam weld number (same as the section number), <i>e.g.</i> , P1S1 translates to Pipe number one and seam weld (or section) number one.							

Table 1. As-Received Pipe Section Dimensions and Construction Detail

Isometric views of the pipe section models can be seen in Figure 3. Not all pipe sections received were coated for corrosion protection, some had some coating and others had none, which resulted in significant corrosion. All sections removed were sent out to a vendor to have that coating removed by media blasting with a fine-grained Silicon-Carbide (SiC) media.



Figure 3 - Isometric view of each modelled pipe section.

The sections removed by plasma cutting were then documented and modelled in the 3D-CAD software modelled, the specimen layout and cut path was designed to maximize the use of available material. Blanks were sectioned for Charpy V-notch (CVN), single-edge bend (SE(B)), single-edge tension (SE(T)), base metal round tensile specimens, all weld-metal (AWM) round tensile specimens, along with net shape full-thickness longitudinal and transverse tensile specimens. Not all specimen types were machined for this study, blanks were retained for potential future research. A total of 19 plasma cut sections were sent to the waterjet vendor. A representative cut plan is shown in Figure 4. All 19 cut plans (including annotations for specific specimen names) are provided in Appendix C. The cut plans were named according to the location of the section, for example P1S1 is a cut plan from pipe section 1 oriented 90 degrees from the seam weld, and P1S1-180 is a section from pipe section 1 oriented 180 degrees from the seam weld. A summary of the test specimens taken from each section is given in Table 2, and more detailed test matrices for specific tests are provided in the following section.



Figure 4 - Example of a specimen sectioning plan sent to the waterjet vendor. This plan is for section P1S1, all dimensions are in inches, more detail may be found in Appendix C. Specimen blanks shown are used for CVN, tensile, SE(B) and SE(T) tests. Samplings for microstructure, chemistry and micro-hardness maps are taken from remaining sprue material.

Section	Microstructure	Chemistry	CVN	Tensile	SE(B)	SE(T)	Hardness
P1S1	SW, BM	SW, BM	SW, BM	SW, BM			SW, BM
P1S1-90	GW, BM	GW, BM	GW, BM	BM			GW, BM
P1S1-180	BM	BM	BM	BM			BM
P1S2	SW, BM	SW, BM	BM	SW, BM			SW, BM
P1S2-90	GW, BM	GW, BM		GW, BM			GW, BM
P1S2-180	BM	BM	BM	BM			BM
P2S1	SW, BM	SW, BM	SW, BM	SW, BM			SW, BM
P2S1-90	GW, BM	GW, BM	GW, BM	BM			GW, BM
P2S1-180	BM	BM	BM	BM			BM
P2S2	SW, BM	SW, BM	SW, BM	SW, BM			SW, BM
P2S2-90	GW, BM	GW, BM	BM	GW, BM			GW, BM
P2S2-180	BM	BM		BM			BM
P3S1	SW, GW, BM	SW, GW,	SW, GW,	SW, GW,	SW	GW,	SW, GW,
		BM	BM	BM		BM	BM
P3S1-90	GW, BM	GW, BM	GW, BM	GW, BM	BM	GW,	GW, BM
						BM	
(P3S1-270)	GW, BM	GW, BM	GW, BM	GW, BM			
P3S1-180	GW, BM	GW, BM		GW, BM	BM	GW,	GW, BM
						BM	
P3S2	SW, BM	SW, BM	BM	SW, BM	BM	BM	SW, BM
(P3S2-90)	GW, BM	GW, BM	BM	GW, BM		BM	GW, BM
(P3S2-270)	BM	BM	BM	BM		BM	BM
(P3S2-180)	BM	BM	BM	BM		BM	BM
P3S3	SW, BM	SW, BM	SW, BM	SW, BM			SW, BM
P3S3-90	GW, BM	GW, BM	BM				GW, BM
P3S3-180	GW, BM	GW, BM	BM				GW, BM

Table 2 - Specimen sectioning summary.

GW = Girth Weld (includes WMC and HAZ, as appropriate)

SW = Seam Weld (includes WMC and HAZ, as appropriate)

BM = Base Metal (includes longitudinal and transverse orientations)

Sections in parenthesis are included in other section cut plans.

Section numbers with -90, -180 and -270 refer to the circumferential section placement with respect to the seam weld (in the "clock" coordinate system common for line pipe, these would be 3 o'clock, 6 o'clock and 9 o'clock respectively).

Specimens for metallographic analysis and indentation were sectioned from the remaining material after water jet cutting. A full layout of all pipe sections with the location of metallographic/indentation samples is included in Appendix D.

# **2.2.** Design of Experiments

## 2.2.1. Chemical Characterization of Base Metal

Accurate chemical characterization of the vintage X100 base metal is critical to understand how the pipe in question compares to historical and modern steels. Optical emission spectroscopy (OES) was used in this study to determine the chemistry of each of the seven pipe sections (P1S1, P1S2, P2S1, P2S2, P3S1, P3S2, and P3S3) in accordance with ASTM E415 [41]. OES specimens were sections from the metallographic test specimens (Appendix D). The pipe specimens were not adequate to cover the OES aperture, so specimens were melted prior to chemical characterization. The test matrix for this task is given in Table 3.

Section	Seam Weld	Girth Weld	Base Metal	<b>OES Chemistry</b>
P1S1	Х		Х	X (BM, SW)
P1S1-90		Х	Х	
P1S1-180			Х	
P1S2	Х		Х	X (BM, SW)
P1S2-90		Х	Х	
P1S2-180			Х	
P2S1	Х		Х	X (BM, SW)
P2S1-90		Х	X	
P2S1-180			Х	
P2S2	Х		Х	X (BM, SW)
P2S2-90		Х	Х	
P2S2-180			Х	
P3S1	Х		X	X (BM, SW)
P3S1-90		Х	Х	
(P3S1-270)		Х	X	
P3S1-180		Х	Х	
P3S2	Х		Х	X (BM, SW)
(P3S2-90)		Х	Х	
(P3S2-270)			Х	
(P3S2-180)			Х	
P3S3	Х		X	X (BM, SW)
P3S3-90		X	X	
P3S3-180		X	Х	

 Table 3 - Test matrix for microstructure and chemical analysis – Specimen micrographs are found in Appendix D.

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#### 2.2.2. Characterization of Microstructure and Chemistry of Vintage and Modern X100 Pipe

The welding process is inherently non-equilibrium the solid-state heating and cooling of the weld pool and HAZs can lead to large gradients in material microstructure and chemistry. These gradients and inhomogeneities will have significant impact on the mechanical properties of the weld zone and should be fully characterized to complement investigations of mechanical properties. In this task, NIST analyzed the microstructure and chemistry of the base metal, HAZs, and weld zones via optical and scanning electron microscopes.

Optical imaging provides qualitative information about gradients in grain size, shape, and morphology across the entire weld area and large areas of base metal. The speed of optical imaging means that full optical images of all metallographic specimens have been collected. The full optical images are large (>500 MB each) so they are provided in the data repository (see Section 1.4). A subset of the optical images is shown in the results section.

Backscattered electron imaging in the scanning electron microscope can provide more quantitative information over the entire weld area. Additionally, electron backscatter diffraction (EBSD) provides localized information about crystallographic texture and grain size.

The test matrix for this task is given in Table 3. Microstructure and chemistry information is necessary for each specific component (seam weld, girth weld or base metal) and orientation of the sectioned pipes, one view is provided for each and is available in the project data repository (see Section 1.4).

#### 2.2.3. Instrumented Charpy Testing and Hardness Mapping of Vintage and Modern X100 Pipe

NIST owns three Charpy reference machines (compliant with ASTM E23 [42]) that help maintain an accurate absorbed energy scale for Charpy machines around the world. Currently, NIST certifies twenty-one standard reference materials (SRMs) that underpin quality control of impact toughness for structural steels. Moreover, NIST is currently working with other national metrological institutes (NMIs) to develop an approach to SRM certification that reduces the measurement bias, with a focus on standardizing the design of instrumented strikers to provide comparable force-displacement data across all types of Charpy machines via a true dynamic calibration procedure. As such, absorbed energy measured under the instrumented impact curve will be traceable to force and time, linking the measurement to more fundamental quantities.

Transition curves for absorbed energy (traditionally known as *KV*) were obtained for base metals of all pipes and sections in the longitudinal (L) orientation, by testing between 8 and 12 third-size Charpy specimens in a range of temperatures encompassing lower shelf, ductile-to-brittle transition region, and upper shelf (between -196 °C and 100 °C). The third-size specimens tested (Figure 5) had a width W = 3.3 mm, which corresponds to 1/3 of the width of a standard Charpy specimen (W = 10 mm), while thickness *B* and length *L* were the same (10 mm and 55 mm, respectively). The notch depth (0.66 mm) also corresponds to 1/3 of the notch depth for a standard specimen (2 mm). From each *KV* transition curve, the values of ductile-to-brittle transition temperature, *DBTT*, and upper shelf energy, *USE*, were calculated. In addition to *KV* values, values of lateral expansion (*LE*) and shear fracture appearance (*SFA*), estimated from the instrumented force/deflection curves, are reported for information only. All tests were instrumented.



Figure 5 – Third-size Charpy specimens used in this investigation.

For the base metals of selected pipes and sections in the transverse (T) orientation and in different clock positions (0°, 90°, 180°), 3 to 4 third-size Charpy specimens were tested at 0 °C. The results obtained in the 90° clock position (P1S1, P2S2, P3S1, P3S2, and P3S3) were compared

with the requirements of API 5L for pipe body of PSL 2 pipes, multiplied by the ratio between sub-size and standard Charpy specimen widths.

For the weld metal and heat-affected zone of each seam weld, between 3 and 6 third-size Charpy specimens were tested at 0 °C. The resulting *KV* values were then compared with the requirement of API 5L for pipe weld and HAZ tests, again multiplied by the ratio between sub-size and standard Charpy specimen widths.

Finally, two third-size Charpy specimens were tested at 0 °C for each girth weld (one in the weld metal and one in the HAZ). Overall, 168 third-size Charpy specimens were tested.

The test matrix for this task is given in Table 4. Data and discussion from the testing are provided in the results section. The full data set is provided in the NIST data repository (see Section 1.4). The number of specimens tested are shown in parentheses: L and T refer to longitudinal specimens and transverse specimens (these are specimen orientations), the notch orientations are perpendicular to the specimen orientation and are not through-thickness but rather notched from the inside-diameter (ID) side of the section.

Section	Seam Weld	Girth Weld	Base Metal
P1S1-0	WMC (3), HAZ (3)	WMC (1), HAZ (1)	L (4), T (4)
P1S1-90			L (4), <b>T (4)</b>
P1S1-180			L (4), T (4)
P1S2-0	WMC (3), HAZ (3)		L (4), T (4)
P1S2-90			L (4), <b>T (4)</b>
P1S2-180			L (4), T (4)
P2S1-0	WMC (3), HAZ (3)	WMC (1), HAZ (1)	L (4), T (4)
P2S1-180			L (4), T (4)
P2S2-0	WMC (3), HAZ (3)		L (4), T (3)
P2S2-90			L (4), <b>T (4)</b>
P3S1-0	WMC (3), HAZ (3)	WMC (1), HAZ (1) [0°]	L (4)
		WMC (1), HAZ (1) [90°]	
P3S1-90			L (4), <b>T (4)</b>
P3S1-180			L (4)
P3S2-0	WMC (3), HAZ (3)		L (4)
(P3S2-90)			L (4), <b>T (4)</b>
(P3S2-180)			L (4)
P3S3-0	WMC (3), HAZ (3)		L (4), T (4)
P3S3-90			L (4), <b>T (4)</b>
P3S3-180			L (4), T (4)
Tests that were com were tested at 0 °C, curves.	pared to the API 5L r except those longitud	equirements are indicated in dinal specimens used to esta	n bold. All specimens ablish the transition

#### Table 4 – Test Matrix for Charpy V-Notch Testing.

Specimens were all subsize (third-size) type due to pipe wall thickness. For full-size Charpy specimens, the minimum average absorbed energy for the pipe body is 54 J (40 ft-lbs) for X100 pipes, whereas the minimum average absorbed energy for welds and HAZ is 40 J (30 ft-lbs).

#### 2.2.4. Tensile Testing of Vintage and Modern X100 Pipe

NIST participated in and conducted research on a modern X100 pipeline steel and girth welds under DOT/PHMSA Research Project DTPH56-07-T-000005, Weld Design, Testing, and Assessment Procedures for High Strength Pipelines [1]. In addition to conducting curved wide plate (CWP) tests, NIST and project collaborators conducted several small-scale mechanical and fracture tests. Small-scale test results have been disseminated in the final reports of the project.

In this current work, NIST conducted similar small-scale tests on the X100 vintage experimental pipe as were conducted in the previous studies. These tests included full thickness base metal tensile tests to determine longitudinal and transverse tensile properties. These tests also included full thickness tensile tests characterizing both seam welds and girth welds.

The complete test matrix (see Table 2) includes single edge-notch bend (SE(B)) and single edgenotch tension (SE(T)) fracture mechanics tests. It is important to note that these fracture mechanics tests were not performed on the pipe prior to putting it into service, therefore no time-history comparisons can be made. To ensure the test results accurately represent material property differences, the standards, methods, instrumentation techniques, and analysis procedures were compared and considered for potential bias in addition to the uncertainty from the tests. The first proposed activity was to review the original test standards, methods, instrumentation techniques and analysis procedures used for testing the vintage and modern X100 steel and girth-welds. The originals are compared to currently used and accepted techniques and procedures to find differences that would influence the comparison of properties.

The pipes and welds were sectioned, and specimens machined per the test matrix. The next activity was to compare test results and determine the material property differences between each pipe section.

The tensile test matrix for this task is given in Table 5. Summary data from the testing is provided in the results section. Complete test records from each specimen are provided in the project data repository (see Section 1.4). The number of specimens tested are shown in parentheses: L and T refer to longitudinal specimens and transverse specimens.

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The sectioning of tensile specimens from pipe sections is illustrated in Figure 6. This figure is reproduced from Figure 5 of the API 5L specification. The L90 specimen is not required for pipe diameters greater than 20 in. Furthermore, longitudinal specimens are not required at all to meet the API 5L line pipe specification. All longitudinal tests were performed for comparative purposes between original material performance data and modern X100 line pipe steel. Girth weld testing in the longitudinal direction is also not a requirement for API 5L.

Round tensile specimens are an optional geometry according to API 5L, however there are two geometry requirements that are impossible to meet with the vintage pipes provided. Firstly, the minimum diameter for a round tensile specimen according to API 5L is 0.25 in (6.35 mm), and this is not possible on a reduced section with a nominal pipe wall thickness of 0.25 in (6.35 mm). Even if a smaller diameter was used, meeting ASTM A370, the API 5L specification requires that specimens in the transverse direction are made from un-flattened pipe. This would only be possible from the larger wall thickness pipes of P3S1 and P3S2 and very limited comparisons would be possible, so the round bar tensile geometry was not used in this study.

Section	Seam Weld	Girth Weld	Base Metal		
P1S1	L (1), T (2) <b>W (1)</b>		L (1)		
P1S1-90			L (3), T (3) <b>T90 (3)</b>		
P1S1-180			L (3), T (3) <b>T180 (1)</b>		
P1S2	L (1), T (3) <b>W (1)</b>		L (1)		
P1S2-90		L (3)	L (3), T (3) <b>T90 (3)</b>		
P1S2-180			L (2), T (3) <b>T180 (1)</b>		
P2S1	L (1), T (1) <b>W (1)</b>		L (1)		
P2S1-90			L (3), T (3) <b>T90 (3)</b>		
P2S1-180			L (3), T (3) <b>T180 (1)</b>		
P2S2	L (1), T (3) <b>W (1)</b>				
P2S2-90		L (3)	⊤ (3) <b>T90 (3)</b>		
P2S2-180			L (2), T (3) <b>T180 (1)</b>		
P3S1	L (1), T (3) <b>W (1)</b>		L (2)		
P3S1-90			L (3), T (3) <b>T90 (3)</b>		
P3S1-180		L (3)	L (3), T (3) <b>T180 (1)</b>		
(P3S1-270)		L (3)	L (3)		
P3S2	L (1), T (3) <b>W (1)</b>		L (3), T (3)		
(P3S2-90)			L (3), T (2) <b>T90 (3)</b>		
(P3S2-180)			L (1), T (3) <b>T180 (1)</b>		
(P3S2-270)			L (3), T (2) <b>T90 (3)</b>		
P3S3	L (2), T (6) <b>W (1)</b>		L (4)		
P3S3-90		L (3)	L (6), T (6) <b>T90 (3)</b>		
P3S3-180			L (6), T (6) <b>T180 (1)</b>		
Numbers in parentheses are the number of specimens tested per section. Bold numbers					

Table 5 – Test Matrix for Tensile Testing.

Numbers in parentheses are the number of specimens tested per section. Bold numbers represent the API 5L minimum requirement. P3S2-90 only has two of the required T90 specimens, however P3S2-270 should be equivalent and has two more specimens to meet the minimum test specimen requirement.



Key

1 W — transverse weld sample, centred on the weld

2 T180 — transverse sample, centred ≈ 180° from the longitudinal weld

3 T90 — transverse sample, centred ≈ 90° from the longitudinal weld

4 L90 — longitudinal sample, centred ≈ 90° from the longitudinal weld

Figure 6 - Schematic illustration of the required specimen orientation and circumferential location of tensile test specimens according to API 5L.

#### 2.2.5. Multiscale Indentation Mechanics of Vintage and Modern X100 Pipe

The hardness profiles of the weld zone were characterized in the R.S. Ryan paper [1] using a Knoop hardness test. This technique clearly revealed a reduced hardness in the HAZ. The following decades have since resulted in dramatic improvements in the spatial resolution of indentation-based mechanics.

In this task, the single Knoop line profile (duplicating R.S. Ryan) is complemented by automated large-area indentation over both girth and seam welds, extending the one-dimensional historical data array into a two-dimensional property map. This allows for direct comparison with the historical data, while introducing greater spatial resolution for characterizing the HAZ to base metal transition zones.

Knoop hardness traces are made across all seam and girth welds. Vickers indentation is used to measure variations in mechanical properties through the pipe wall thickness. Additionally, Vickers indentation is used to map properties across entire girth and seam welds. The Vickers indentation test results in a smaller, more symmetric indent allowing for higher resolution property mapping over large areas.

Instrumented nano-indentation (using a Berkovich indentation geometry) is used to measure the mechanical property of materials at the nanometer scale. Nano-indentation results in a much smaller plastically deformed zone and much higher precision in lateral positioning, allowing for spatial resolution 100x to 1000x better than Vickers or Knoop indentation. In this case, instrumented nano-indentation is used to measure how variations in the material microstructure (specifically near the internal and external surfaces of the pipe) impact the localized mechanical properties.

The test matrix for this task is given in Table 6. Representative data from the Knoop, Vickers, and nano-indentation testing is provided in the results section. Complete test data are included in the data repository (see Section 1.4).

Section	Seam Weld	Girth Weld	Base Metal
P1S1	Х		Х
P1S1-90		Х	
P1S2	Х		Х
P1S2-90		Х	
P2S1	Х		Х
P2S1-90		Х	
P2S2	Х		Х
P2S2-90		Х	
P3S1	Х		Х
P3S1-90		Х	
(P3S1-270)		Х	
P3S1-180		Х	
P3S2	Х		Х
(P3S2-90)		Х	
P3S3	Х		Х
P3S3-90		Х	
P3S3-180		Х	

Table 6 - Test matrix micro-hardness mapping of welds.

# 2.3. Metallography

# 2.3.1. Metallographic Preparation

After separation from their host plate, the metallographic samples were sectioned using a highspeed saw. The saw and the specimen were water-cooled during cutting to provide lubrication

and prevent any significant temperature rise in the specimen being sectioned. No specific corrosion protection was provided during this step, as all specimens would be polished following sectioning. The metallographic samples were sectioned to a major dimension of less than 31 mm to allow for mounting into phenolic resin.

Specimens were then mounted in a thermosetting, electrically conductive phenolic resin using a heated mounting press.

Damage from the high-speed saw was removed using diamond-embedded disks, moving to finer grits during polishing, including 240 grit, 320 grit, 480 grit, 600 grit, 800 grit, and 1200 grit. Following grinding with the diamond disks, specimens were polished using diamond slurry decreasing in grit size including 9  $\mu$ m, 6  $\mu$ m and 3  $\mu$ m. All specimens were subsequently polished with the 1  $\mu$ m diamond slurry until all scratches from previous polishing steps were removed.

For electron backscatter diffraction and nano-indentation, the specimens were polished using a 0.05  $\mu$ m colloidal silica suspension with a vibratory polisher for up to four hours. Polishing for more than four hours was found to produce surface relief in the metallographic specimens.

#### 2.3.2. Optical Microscopy

Specimens for optical microscopy re-polished using the preparation steps listed previously and were etched using a 2.5% Nital etchant (5 ml 50% nitric acid solution to 100 ml methanol) to reveal the grain structure. Specimens were submersion etched for approximately 30 seconds, until the polished surface clouded over, and the bulk grain structure was visible. Specimens were immediately washed with isopropanol to prevent corrosion.

Etched metallographic specimens were imaged with a digital optical microscope to provide a bulk overview of the microstructure. Specimens were then imaged in bright field with an inverted metallographic microscope. Images were collected at a magnification of 10x (0.61 µm pixel size) with an image size of 2048 x 2048 pixels. The open-source micro-manager software [43] was used to image over large areas and stitch the resulting images.

A portion of the optical microscope images are included in this report, but the images are very large (>200 MB per image), so the full data set is provided in the data repository (see section 1.4).

#### 2.3.3. Scanning Electron microscopy

Backscattered electron images were collected to provide higher resolution imaging of the base metal, weld, and heat affected zone microstructures. Images were collected with an accelerating voltage of 5 kV, a beam current of 1.3 nA. Dwell time was adjusted as appropriate to produce acceptable single-to-noise ratio. An automated large-area imaging and image stitching software package was used to collect and stitch images over large areas.

In addition, electron backscatter diffraction (EBSD) was used to measure microstructure metrics in welds, heat-affected zones, and base materials. The scanning electron microscope was operated under the following conditions on samples tilted 70 degrees: 30 kV accelerating voltage, 120 um aperture, and a 19 mm working distance. Large areas were surveyed using a multi-tile method where a given tile was approximately 450 µm x 450 µm in size and the entire row of stitched tiles spanned a length of 8 mm. These EBSD maps were recorded using a step size of 750 nm. Smaller areas were further analyzed in the base metal using a 250 nm step size during acquisition of a single tile.

#### 2.4. Tensile Testing

Testing according to API 5L requires that ASTM A370 be followed as the standard test method, ASTM A370 references ASTM E8. While ASTM A370 provides an overview and references ASTM E8 for detailed procedures, ASTM E8 contains the in-depth guidelines required to perform tension testing accurately and consistently. Despite specimens being curved, the standard application will be for plate-type specimens having a reduced section width of 1.5 in (38.1 mm) and a gauge length of 2 in (50.8 mm). Values stated in in-pound units are regarded as the standard. Units converted from inch-pound to SI are not required for the standard, furthermore in-pound units were the original units used when the vintage X100 pipe sections were manufactured. The plate-type specimen dimensions relevant to this study follow the *Rectangular Tension Test Specimens* fully described in Figure 3 of the ASTM A370 standard (see also Figure 1 of the ASTM E8 standard). Allowable deviations to the specimen geometry are related to the length and width of the grip section (grip-tab). Gauge marks for the purpose of measuring elongation after fracture are replaced with welded studs, described below in section 2.4.3.

#### 2.4.1. Specimen Geometry and Measurements

Full-wall-thickness specimens were waterjet cut from each pipe section; the specimens were dogbone shaped where the reduced section length was greater than 8 times that of the thickness. All specimens had the same nominal shape, shown in Figure 7. However, the waterjet nozzle was always perpendicular to the working surface and not perpendicular to the tangent point on the surface of the pipe section. This resulted in specimens with parallel edges, but not necessarily perpendicular to the inside diameter or outside diameter surfaces of the pipe section. The crosssectional area, for stress calculation, was taken as the edge-to-edge width multiplied by the average thickness of the specimen (pipe wall), measured for each specimen. The thickness was measured with a digital micrometer equipped with a flat anvil (OD side) and a ball end anvil (ID side). Three thickness measurements along the length of the specimen were taken and averaged. The edge-to-edge width was the average of three measurements along the reduced section using digital calipers.



Figure 7 - Nominal shape and dimensions of full-wall-thickness tensile specimens. All dimensions are in inches.

#### 2.4.2. Flattening Procedure

Specimens tested that were transverse to the longitudinal axis of the pipe sections required flattening prior to testing. The flattening procedure for these specimens used a four-point bend fixture. Specimens were placed into the fixture and were loaded and moved incrementally to reduce the occurrence of reverse-bending the specimens. The four-point bend fixture is shown in Figure 8. Each specimen required manual adjustments to the incremental loads applied, since a standard load or displacement value for each successive loading did not lead to the desired outcome.



Figure 8 - Flattening fixture used for transverse tensile specimens. Four-point bend arrangement with a lower span of 4 in and upper span of 2 in.

The procedure for each incremental loading began with the specimen grip-tab edge close to the center of one of the bottom span rollers as shown in Figure 8. After loading and unloading, the specimen was shifted so that it was centered within the upper and lower spans and was loaded and unloaded again. The specimen was then shifted again so that the opposite grip-tab was flattened, with a similar alignment to the first loading. The specimen was shifted within these three positions with progressive loading and unloading, until the center (reduced gage section)

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was flattened. Flatness of the gage section was verified for each specimen using a ground steel reference plate and feeler gauges (within 0.5 mm). The ends of the specimens, specifically, nearest the grip-tab edges were the least flat section resulting from this method.

This method required some craft to obtain the best possible flatness in the gage section without inducing a reversal in curvature, and subsequent correction. The method was effective on base metal and welded specimens; however, it was more difficult to demonstrate flatness on the welded specimens due to the root pass weld reinforcement.

The remaining curvature of the end tabs presented a possible source of bending in the specimen at the beginning of the test. One of the specimens was instrumented and loaded into the hydraulic grips while data was recorded to capture any bending strain induced due to the gripping process. The procedure was to install and grip the specimen in the upper hydraulic grip, install the extensometer, and zero (tare) the force and extensometer signals while in displacement control. Then switch to force control (set at zero force) and apply gripping pressure on the lower grip-tab while collecting data from the process. Examining the data from this process showed that less than 0.2 % strain was imposed on the specimen as a combined result of a bending moment induced by the gripping processes on the curved grip-tabs and the change in machine displacement to accommodate the zero-force command. This amount of strain is above the signal noise floor but is not large enough to require a procedure change. Furthermore, the posttest analysis procedure for each test includes slack compensation that eliminates non-linear response at the beginning of the test.

#### 2.4.3. Specimen Preparation

Each specimen was measured and scribed for the installation of a threaded stud. The threaded studs were welded to the ID surface of the specimens with a capacitive discharge stud welder. A cam-like fixture with a hardened steel pin was installed on each stud to interface with the extensometer. A post-test photo of a specimen is shown in Figure 9, illustrating the alignment scribe lines and showing the welded studs. A specimen loaded into the hydraulic grips is shown in Figure 10, along with a photo of the extensometer installed on the specimen.



Figure 9 - Post-test photo of a tensile specimen.



Figure 10 - Full wall thickness tensile specimens were tested in a servo-hydraulic universal test frame and were gripped in hydraulic grips (left). A close-up view of the gage section of the specimen on the ID side shows how the extensometer is attached to the specimen prior to testing (right).

### 2.4.4. Tensile Testing Procedure

All instruments used during the test procedure were calibrated according to the manufacturer's specification and appropriate ASTM Standards. Each tensile test was conducted according to the same machine control profile. After measurement/layout and stud welding procedures (see above section), each specimen was tested according to the following procedure:

- 1. Measure specimen thickness and gage length between pins and input into the summary spreadsheet.
- 2. On gage pins, add a nut, clip gage fixture, and another nut tighten when aligned.
- 3. Insert tensile specimen into top hydraulic grip, center it, then tighten w/ hydraulic pressure.
- 4. Raise bottom grip in displacement mode.
- 5. Zero the force signal.
- 6. Go to force control and immediately close the bottom hydraulic grip.
- 7. Apply a pre-load to the specimen using manual command 0.1 kip in tension.
- 8. Install the extensometer and zero the signal.
- 9. Remain in force control and release the manual command.
- 10. Click New Specimen in the program "Flat Tensile Test" and type the specimen ID.
- 11. Click the start button, fill in the user input window, click Ok.
- 12. While the test runs, measure the next specimen's dimensions and update the summary spreadsheet.
- 13. At the completion of the test, click new specimen and switch to displacement control.
- 14. Remove the extensometer.
- 15. Release the bottom hydraulic grip remove the specimen half.
- 16. Release the top hydraulic grip remove the specimen half.
- 17. Remove the nuts and clip gage fixtures from the welded studs.
- 18. Measure total elongation and input into the summary spreadsheet.

Each specimen was tested to failure at room temperature using a displacement-controlled rate

of 0.05 in/min.

### 2.4.5. Tensile Analysis Procedure

The data record for each specimen was analyzed using a spreadsheet template with data reduction and analysis algorithms developed by NIST for this program. This ensured that each specimen was analyzed using the same procedure with similar variable adjustments.

The following generalized procedure uses the spreadsheet and requires some level of engineering judgement; the number of specimens analyzed did not warrant the next level of programming to automate the analysis.

- 1. Open the tensile analysis spreadsheet template.
- 2. Navigate to the (next) specimen folder and open the specimen.dat file.
- 3. Copy-Paste the tab-delimited columns from the specimen.dat file into the Raw Data worksheet of the analysis spreadsheet.
- 4. Examine the plot shown on the Raw Data worksheet and determine the last significant data point.
- 5. Delete all data rows past that last significant data point.
- 6. On the Reduced Data worksheet adjust the number of rows to match the raw data.
- 7. Examine the Slack Plot and adjust the range to match the best linear portion of the curve.
- 8. Using the Goal Seek function (on cell J21), find the 0.2 % offset yield stress.
  - a. Adjust the strain range to ensure that the yield strain is within the range.
  - b. Iterate as needed.
- 9. Copy the specimen results into the summary spreadsheet.
- 10. Save the file with the specimen ID in the appropriate folder save the analysis file as the next specimen ID and repeat the steps starting with number 2.

The spreadsheet automatically calculates the engineering stress, engineering strain, and slackcompensated engineering strain. The slack-compensated engineering strain is dependent on the user defined linear range (taken as a percentage of the UTS). This is the same linear range used to calculate the elastic modulus. Slack compensation can affect the 0.2 % offset yield, the stress at 0.5 % strain, as well as the strain at the ultimate tensile strength (UTS), also referred to as uniform elongation. The source of slack can include straightening of curved specimens, noise in the extensometer data, or non-linearities associated with the extensometer seating on the specimen gage pins.

A representative Slack Plot is shown in Figure 9. Here, the difference between the original straindependent data and the slack-compensated data is less than 0.13 % for this specimen; some specimens exhibited more signal noise and non-linearity at the beginning of the test than this

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one. The maximum difference between the original and slack-compensated strain-dependent data for all the tests was 2.3 %. The horizontal lines in Figure 11 define the upper and lower bounds over which the modulus is calculated. The fitting coefficients of that line are used to calculate the offset strain to compensate for slack. The vertical line is the 0.5 % strain line, and the corresponding stress is determined as the intersection of that line with the stress-strain curve. The 0.2 % offset line has the same slope as the calculated modulus, and the stress is determined as the intersection of that line with the stress is



Figure 11 - Stress-strain curve of specimen P4 (from P3S2), illustrating the Slack Plot used in the data analysis spreadsheet. The slack compensation for this specimen was minimal, resulting in a uniform elongation difference of 0.003 %.

Only the data that are relevant to this comparative study is presented. According to API 5L, the yield stress (0.2 % offset method), the yield stress (defined as the stress at 0.5 % strain), the

ultimate tensile strength (UTS), yield to tensile ratio, uniform elongation, and seam weld strength are the only tensile properties of concern.

The tensile requirements for X100Q PSL 2 pipe according to API 5L are given in Table 6. The specified minimum elongation is given by the following equation:

$$A_f = C \frac{A_{xc}^{0.2}}{U^{0.9}}$$

where C is 625,000,  $A_{xc}$  for all specimens tested in this study is the cross-sectional area of the specimen measured to the nearest 0.01 in<sup>2</sup> and U is the minimum tensile strength which is 110,200 psi.

		Pipe l	Body Prope	rties			Seam Weld
Yield Strength (psi)		UTS (psi)		Y/T (ul)	A <sub>f</sub> (%) min		UTS (psi)
min	max	min	max	max	0.25 in	0.5 in	min
100,100	121,800	110,200	143,600	0.97	13.7	17	110,200
Yield Strer	ngth (MPa)	UTS (	MPa)		A <sub>f</sub> (%	) min	UTS (MPa)
min	max	min	max		6.35 mm	12.7 mm	min
690	840	760	990		13.7	17	760

Table 7 - Tensile property requirements for X100M or X100Q – excerpt from API 5L.

There is a caveat in API 5L for the yield to tensile (Y/T) ratio for grades greater than X90, where instead of the yield strength being defined as the stress at 0.5 % strain, the yield stress for this ratio is the 0.2 % offset yield stress.

# **2.5.** Instrumented Charpy Testing

Charpy impact tests on third-size specimens extracted from base metals, weld metals, and heat affected zones were conducted on a large-capacity (950 J) machine equipped with an 8 mm<sup>1</sup> instrumented striker. The velocity at impact was 5.47 m/s.

The energy absorbed at specimen fracture (absorbed energy, *KV*) was measured by means of a digital encoder, based on the fall, and rise angles of the hammer and accounting for windage and

<sup>&</sup>lt;sup>1</sup> 8 mm indicates the radius of the striking edge.

friction losses. As ancillary information, the following was measured and reported for every specimen tested:

- lateral expansion, *LE* (mm): combined height of the shear lips generated by plastic deformation of the sample during fracture, measured by means of a caliper; and
- (estimated) shear fracture appearance, *SFA<sub>est</sub>* (%): percentage of ductile (shear) fracture surface.

The latter parameter, SFA<sub>est</sub>, was calculated from the instrumented force-displacement curve of

each test performed (see an example in Figure 12), using the following equation:

$$SFA_{est} = \left[1 - \frac{F_{iu} - F_a}{F_m + 0.5(F_m - F_{gy})}\right] \times 100$$
 (1)

where in Figure 12:

- *F<sub>iu</sub>* = force at unstable crack propagation
- *F<sub>a</sub>* = crack arrest force
- *F<sub>m</sub>* = maximum force
- $F_{gy}$  = force at general yield.

Eq. (1) is one of four formulae reported in the ASTM E2298 and ISO 14556 standards. However, this is the one adopted at NIST for *SFA* estimation, based on results published in [44].



Figure 12 - Example of instrumented force/deflection curve for a test in the ductile-to-brittle transition region.

For some tests, instrumented data were not recorded due to a malfunction of the acquisition system. In other instances (particularly at the lowest temperatures and for extremely brittle specimens), the analysis of the instrumented curve was extremely difficult, due to the low levels of force and the very pronounced dynamic oscillations that were superimposed onto the actual test record, which rendered almost impossible to determine the characteristic values of force shown in Figure 12.

For each set of tests performed on the base metal of a specific pipe/section in longitudinal (L) orientation, absorbed energy values, *KV*, were fitted as a function of test temperature using the well-established hyperbolic tangent (TANH) regression model [37]:

$$KV = \frac{LSE + USE}{2} + \frac{USE - LSE}{2} \cdot tanh\left(\frac{T - DBTT}{C}\right)$$
(2)

where:

- *T* is the temperature (°C),
- LSE (lower shelf energy) is the asymptotic value that the curve tends to as T decreases (J)
- USE (upper shelf energy) is the asymptotic value that the curve tends to as T increases (J)
- C is the half-width of the transition region between lower and upper shelf (°C)
- *DBTT* is the ductile-to-brittle transition temperature (°C), corresponding to the point where *KV* = (*LSE*+*USE*)/2

The regression curve obtained is denominated *transition curve* (example in Figure 13), and the main material parameters extracted from it are *DBTT* and *USE*. In the analyses conducted here, *LSE* was set at the minimum recorded value of absorbed energy.



Figure 13 - Example of absorbed energy transition curve for a low-strength steel.

### 2.6. Indentation Testing

#### 2.6.1. Microhardness Indentation

Following mounting and metallographic preparation (including final polishing with 1  $\mu$ m diamond slurry), specimens were placed in an automated microhardness tester. Both Vickers and Knoop indentations were performed with an indenter load of 500 grams and a dwell time of 13 seconds. Indentations were measured automatically using the microhardness tester software.

Vickers indentation was performed across the thickness of the pipe base metal to establish the degree of hardness variation that may exist due to potential through-thickness micro-structural gradients. Weld cross sections (perpendicular to the weld axis) were mapped with Vickers indentation to measure the hardness across the girth or seam weldments, including pipe steel base metal, heat affected zone (HAZ) and weld metal regions.

Knoop hardness indentation traces were performed across weld cross sections to recreate the data presented by R. S. Ryan [1].

#### 2.6.2. Nano-indentation

Following mounting and metallographic preparation (including final polishing with colloidal alumina), specimens were placed on the stage of the nanoindenter. The nanoindenter was equipped with a precision stage and a load transducer, allowing for instrumented indentation and measurement of local hardness and elastic modulus.

Nano-indentation was performed using a Berkovich geometry indenter tip and a quasistatic load function with a maximum load of 5000  $\mu$ N. Load was applied over 15 seconds, held at the maximum load for 5 seconds, and then unloaded over 15 seconds. Elastic modulus was determined from the loading portion of the force-displacement curve and the hardness was determined based on the known (assumed) tip geometry and the maximum displacement upon loading.

Indentation was performed across a portion of the thickness of the pipe to detect any degree of variation that may exist due to potential through-thickness micro-structural gradients. Additionally, indentations were performed along the internal surface of the pipe to investigate any variation or degradation in mechanical properties that may eventually be attributed to long-term exposure to natural gas, but further research is necessary to explore this possibility.

### 2.7. Results

### 2.7.1. Material Chemistry

One (or more) specimens of base metal from each pipe section (P1S1, P1S2, P2S1, P2S2, P2S3, P3S1, P3S2) were characterized by optical emission spectroscopy (OES), which included meltdown of most specimens as the pipe wall thickness was too thin to fully cover the OES aperture. The chemical characterization results, which were critical in the efforts to identify the pipe in the historical literature, are provided in Table 8. Note that specimen OM2 is from a girth weld connecting pipe specimens P3S1 and P3S2.

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	с	s	Р	Si	Cr	Ni	Mn	Cu	Мо	Nb	Ті	AI	v	Co	w	Sn	Fe
AM1 (P1S1)	0.24	0.02	0.016	0.04	0.05	0.02	1.21	0.03	0.1	<0.01	<0.01	0.01	0.08	0.01	0.01	0.01	Bal
AM2 (P1S1)	0.12	0.024	0.012	0.15	0.04	0.03	0.62	0.03	0.45	<0.01	0.01	<0.01	0.02	<0.01	<0.01	<0.01	Bal
DM3 (P1S2)	0.19	0.018	0.010	0.31	0.02	0.02	1.33	0.04	0.01	<0.01	0.01	0.09	<0.01	<0.01	0.01	<0.01	Bal
GM2 (P2S1)	0.25	0.02	0.011	0.27	0.02	0.02	1.24	0.03	0.01	<0.01	0.01	0.07	<0.01	<0.01	<0.01	<0.01	Bal
JM1 (P2S2)	0.24	0.017	0.010	0.29	0.02	0.02	1.31	0.03	0.01	<0.01	0.01	0.08	<0.01	<0.01	<0.01	<0.01	Bal
MM3 (P3S1)	0.22	0.012	0.011	0.03	0.05	0.02	1.21	0.04	0.01	<0.01	<0.01	0.01	0.08	<0.01	<0.01	<0.01	Bal
OM1 (P3S2)	0.21	0.016	0.01	0.27	0.02	0.03	1.23	0.03	0.01	<0.01	0.01	0.07	<0.01	<0.01	<0.01	<0.01	Bal
OM2 (P3S2, GW)	0.13	0.021	0.018	0.51	0.11	0.63	1.19	0.18	0.12	<0.01	0.01	0.02	<0.01	0.01	0.01	0.01	Bal
RM1 (P3S3)	0.19	0.015	0.009	0.28	0.02	0.02	1.29	0.04	0.01	<0.01	0.01	0.08	<0.01	<0.01	<0.01	<0.01	Bal

#### Table 8 - Chemical composition of vintage X100 pipeline steels from optical emission spectroscopy.

### 2.8. Microstructure Analysis

#### 2.8.1. Optical microscopy and BSE imaging

An example of both SEM backscatter imaging on polished specimens and optical bright field imaging on etched specimens is shown in Figure 14. The provided optical and scanning electron images provide a representation of the base metal microstructure, which is consistent through all pipe sections. It should be noted that the optical images provide information like the backscattered images, but the optical image collection time is approximately 1% of the required time to collect a large-area scanning electron image.

Figure 15 illustrates the various microstructures present in and around a girth weld. Inset images include the interface between the weld metal and heat affected zone (red), the microstructure of the weld metal (green), the fine-grained heat affected zone (blue), and the pipe base metal (orange). Lastly, Figure 16 shows a bright field optical image of a seam weld, the heat affected zone, and the surrounding base metal, showing the consistent grain size between the three distinct regions.

All raw imaging data is provided in the project data repository (see Section 1.4).



Figure 14 – Scanning electron microscope backscatter images (EM3 RTT, EM3 ATT, EM3 PV) and optical bright field images (AM1 RTT, AM1 ATT, AM1 PV) demonstrating the typical microstructure of the pipeline base metal.



Figure 15 – Scanning electron microscope backscatter images illustrating the typical microstructure across the regions of a girth weld (EM1 ATT Weld P1G1).



Figure 16 – Optical image of seam weld illustrating the consistent grain size between the weld metal, heat affected zone, and base metal (AM2 ATT).

### 2.8.2. Electron backscatter diffraction

Samples from selected pipe sections, welds, and orientations were prepared for further analysis using diffraction-based techniques in the SEM. A summary of the three orthogonal views is depicted in Figure 17 where a perimeter girth weld is used in that example. In many cases, the welding process ranged from automated to semi-automated, and manual methods were also employed. Between the welded regions and the joined base metals are heat-affected zones (HAZ) where, in some locations, the microstructure morphology resembles the base metal, but certain characteristics such as local misorientation and grain size tend to differ from the base metal. In Figure 18, there are effectively two HAZ regions where the HAZ closest to the weld is characterized by an extremely refined grain structure, higher kernel misorientation, and greater grain orientation deviation as compared to the weld. The HAZ region closest to the base metal also showed evidence of grain refinement compared to the base metal but was not as drastic of a difference as depicted in the HAZ closest to the weld, evidenced in both the room temperature alpha-Fe grains as well as the parent austenite grains. The HAZ closest to the base

A free copy of this report can be obtained from: https://doi.org/10.6028/NIST.IR.8535 metal also contained the lowest average kernel misorientation and orientation deviation. The average grain reference orientation deviation (GROD) value in degrees of orientation deviation respectively changed from ~5 ° in the weld to ~4 ° in the HAZ near the weld to ~2 ° in the HAZ near the base metal and back to approximately ~4 ° in the base metal. These same trends are visible in the other orthogonal view, depicted in Figure 19. Regardless of the semi-automated girth weld shown in Figure 18 or the manual girth weld shown in Figure 20, the trends of a duality in HAZ regions exists. This is important to note since tensile specimens excised with a tensile direction parallel the horizontal direction of Figure 18 and Figure 20 fractured at the fusion line . The manual girth weld (location EM1) shown in Figure 20 also contains a columnar microstructure (grain growth is parallel to the highest thermal gradient and varies spatially) in the weld. The manual girth weld also contained large spherical gas pores in the weld.



Figure 17 - Schematic of the three orthogonal views (PV, RTT, and ATT) characterized with scanning electron microscopy.



Figure 18 - a) An optical image of a girth weld, HAZ, and base metal from location KM2, depicted by an axial view through the thickness (ATT) where a white rectangle indicates the location of a multi-tile large-area EBSD map. The b) kernel average misorientation c) grain reference orientation deviation, and d) inverse pole figure maps generated with EBSD while indexing body-centered cubic alpha-Fe are shown above. A child-to-parent reconstruction was performed and a plot of the e) inverse pole figure map of the face-centered-cubic gamma-Fe phase is shown.



Figure 19 - a) An optical image of a girth weld, HAZ, and base metal from location KM2, depicted by a plan view of the surface (PV) where a white rectangle indicates the location of a multi-tile large-area EBSD map. The b) kernel average misorientation c) grain reference orientation deviation, and d) inverse pole figure maps generated with EBSD while indexing body-centered cubic alpha-Fe are shown above. A child-to-parent reconstruction was performed and a plot of the e) inverse pole figure map of the face-centered-cubic gamma-Fe phase is shown.



Figure 20 - a) An optical image of a girth weld, HAZ, and base metal from location EM1, depicted by an axial view through the thickness (ATT) where a white rectangle indicates the location of a multi-tile large-area EBSD map. The b) kernel average misorientation c) grain reference orientation deviation, and d) inverse pole figure maps generated with EBSD while indexing body-centered cubic alpha-Fe are shown above. A child-to-parent reconstruction was performed and a plot of the e) inverse pole figure map of the face-centered-cubic gamma-Fe phase is shown.

The base metal was analyzed in three orthogonal views to understand the influence of surface conditions on the microstructure present in these pipe sections. In Figure 21, a multi-tile map was recorded on the ATT view from the outer diameter of the pipe towards the interior base metal. Within the first 200 microns of the outer diameter, a refined grain structure exists and is likely caused by processing routines and environmental effects over time. The refined portion tends to have more randomly oriented grains, lower grain orientation deviation and a smaller grain size. When all three orthogonal planes were analyzed, the trend remained. Figure 22 and Figure 23 respectively quantify the orientation and grain size metrics of the ferritic grains observed with EBSD. The outer diameter surface of the pipe trended towards having a low grain orientation spread of ~ 1°, whereas the interior microstructure consistently showed a grain orientation spread (GOS) value of ~ 2.5°. When comparing grain boundary misorientations for any given field of view, a bi-modal distribution of boundary types was observed to contain a high number fraction of low-angle grain boundaries (0° to 5°) and a broad distribution of high-angle grain boundaries (greater than 15°). Histograms are provided as the density of boundaries in a map of these fields of view would be difficult to discern. The cumulative

percentages of low-angle grain boundaries (0° to 5°) in the ATT, RTT, and PV\_OD views are respectively 57%, 58%, and 42% whereas high-angle grain boundaries (15° to 180°) in the ATT, RTT, and PV\_OD views of the base metal were respectively 38%, 38%, and 54%. The grain size diameter of the ferritic grains near the pipe surface was less than 2 microns whereas the grain size of the interior of the pipe trended more towards 7 microns.



Figure 21 - a) An optical image of base metal from location EM3, depicted by an axial view through the thickness (ATT) where a white rectangle indicates the location of a multi-tile large-area EBSD map from the outer diameter to the interior of the pipe base metal. The b/c/d) child alpha-Fe phase, present at room temperature, and e/f/g) parent gamma-Fe phases are shown side-by-side. Specifically, the b/e) inverse pole figure maps, c/f) grain reference orientation deviation maps, and d/g) grain size diameter maps are provided to show specific changes in microstructure from left (pipe outer diameter) to right (interior of the base metal).



Figure 22 - Single-tile small-area EBSD maps were recorded from all three orthogonal views (ATT, RTT, and PV\_outer diameter) of the base metal at location EM3. The a) inverse pole figure maps, b) grain orientation spread, and c) misorientation histograms provide quantitative differences between the microstructure at the surface of the pipe (PV\_outer diameter) and the interior microstructure.



Figure 23 - Single-tile small-area EBSD maps were recorded from all three orthogonal views (ATT, RTT, and PV\_outer diameter) of the base metal at location EM3. The a) grain size diameter maps and b) grain size diameter histograms provide quantitative differences between the microstructure at the surface of the pipe (PV\_outer diameter) and the interior microstructure.

### 2.9. Tensile Testing

The results presented in Table 9 are based on the requirements of API 5L referenced in Table 7. The data are organized based on the orientation and location of the tested specimens. In cases where multiple specimens were tested, the average value is presented, and the number of specimens tested are indicated by the number in parentheses next to the section identifier (see also Table 5). Sections with multiple specimens are reported as the average with the standard deviation, seam weld specimen for P2S1 is reported with the observed value of stress with an estimated combined uncertainty of 1.06 %.

		Pipe Bo	dy Results			Seam Weld
Section	YS <sub>0.2 %</sub>	YS0.5 %	UTS	Y/T	A <sub>f</sub>	UTS
	ksi	ksi	ksi	ul	%	ksi
	(MPa)	(MPa)	(MPa)			(MPa)
	(IVII d)	(IVII d)	(ivii d)			137 9 + 0 9
P1S1 (2)						$(951 \pm 6)$
D161 00 (2)	112.8 ± 1.7	107.1 ± 2.4	135.2 ± 0.4	0.83 ± 0.01	18.1 ± 0.8	(,
P151-90 (3)	(778 ± 12)	(738 ± 17)	(932 ± 3)			
D464 400 (2)	114.0 ± 0.6	104.2 ± 1.0	135.7 ± 0.4	0.84 ± 0.00	19.1 ± 0.9	
P151-180 (3)	(786 ± 4)	(718 ± 7)	(936 ± 3)			
P1S2 (3)						140.3 ± 0.3
F 132 (3)						(967 ± 2)
P152-90 (3)	113.4 ± 0.7	108.7 ± 0.7	139.1 ± 0.1	0.82 ± 0.00	18.3 ± 0.4	
1 132-30 (3)	(782 ± 5)	(749 ± 5)	(959 ± 1)			
P152-180 (3)	119.4 ± 6.7	116.2 ± 13.2	138.3 ± 0.4	0.86 ± 0.05	18.2 ± 0.5	
1 132-100 (3)	(823 ± 46)	(801 ± 91)	(954 ± 3)			
P2S1 (1)						138.6 ± 1.5
1231(1)						(956 ± 10)
P2S1-90 (3)	115.4 ± 1.1	110.3 ± 3.9	135.6 ± 2.2	0.85 ± 0.01	18.6 ± 0.6	
1231 30 (3)	(796 ± 8)	(760 ± 27)	(935 ± 15)			
P2S1-180 (3)	114.6 ± 1.6	109.5 ± 3.5	135.9 ± 0.6	0.84 ± 0.01	18.6 ± 0.3	
1231 100 (3)	(790 ± 11)	(755 ± 24)	(937 ± 4)			
P2S2 (3)						139.9 ± 1.1
1 202 (0)			1			(965 ± 8)
P2S2-90 (3)	112.4 ± 2.4	98.0 ± 3.4	136.3 ± 0.3	0.82 ± 0.02	15.9 ± 0.6	
0_ 00 (0)	(775 ± 17)	(676 ± 24)	(940 ± 2)			
P2S2-180 (3)	114.9 ± 2.7	106.8 ± 6.1	137.3 ± 0.3	0.84 ± 0.02	16.7 ± 0.7	
000 (0)	(792 ± 19)	(736 ± 42)	(947 ± 2)			
P3S1 (3)						$90.9 \pm 1.3$
		612+08	96.1 + 0.7	0.60 ± 0.01	$21.0 \pm 0.0$	$(627 \pm 9)$
P3S1-90 (6)	$59.8 \pm 0.4$	$61.2 \pm 0.8$	86.1±0.7	0.69 ± 0.01	31.8±0.9	
	$(412 \pm 3)$	(422±6)	$(594 \pm 5)$	0.60 ± 0.00	22.7 + 0.0	
P3S1-180 (3)	$59.7 \pm 0.1$	$(1.5 \pm 0.2)$	$(E07 \pm 1)$	0.09 ± 0.00	52.7 ± 0.0	
	(412 ± 1)	(424 ± 1)	(597 ± 1)			02 2 + 0 9
P3S2 (3)						$95.5 \pm 0.8$
	65.1 ± 3.1	65.8 ± 1.3	89.8 ± 0.2	0.72 ± 0.03	30.0 ± 1.0	(0+3 ± 0)
P3S2-90 (3)	(449 + 21)	(454 + 9)	(619 + 1)			
	65.6 ± 1.7	67.1 ± 1.4	89.2 ± 1.4	$0.74 \pm 0.01$	30.9 ± 0.4	
P3S2-180 (3)	(452 ± 12)	(463 ± 10)	(615 ± 10)			
		(				134.7 ± 1.0
4323 (2)						(928 ± 7)
	108.7 ± 4.7	99.8*± 9.2	130.4 ± 0.9	0.83 ± 0.03	19.6 ± 0.6	. ,
4323-90 (6)	(749 ± 32)	(688 ± 63)	(899 ± 6)			1
	106.2 ± 2.6	97.7*± 3.8	131.3 ± 0.6	0.81 ± 0.02	19.7 ± 0.6	
4323-180 (6)	(732 ± 18)	(674 ± 26)	(905 ± 4)			

#### Table 9 - Tensile testing results required by API 5L.

• Numbers in parenthesis after the section label indicate the number of specimens tested and included in the average and standard deviations given.

• Y/T is the ratio between the yield strength and the ultimate tensile strength.

### 2.10. Instrumented Charpy Testing

### 2.10.1. Absorbed energy transition curves for base metals - Longitudinal Direction

The results obtained from third-size Charpy specimens for the obtainment of KV transition curves are provided in Tables 9 to 15. The tables also include values of lateral expansion and estimated shear fracture appearance.

Specimen ID	т (°С)	<i>к</i> (J)	<i>LE</i> (mm)	SFA (%)	B/FB/NB <sup>2</sup>
C10-L2	-196	0.4	0.06	N/A <sup>3</sup>	В
B15-L3	-150	0.9	0.00	N/A	В
C10-L4	-135	5.8	0.08	28	В
A3-L3	-125	5.4	0.04	N/A	FB
C10-L3	-100	5.7	0.12	N/A	FB
B15-L4	-85	10.2	0.21	N/A	FB
B15-L2	-75	13.9	0.17	N/A	FB
A3-L2	-50	11.9	0.14	N/A	FB
A3-L1	-25	12.3	0.24	100	FB
B15-L1	0	8.6	0.24	100	FB
C10-L1	21	10.5	0.21	100	FB
A3-L4	100	9.3	0.24	100	FB

Table 10 - Third-size Charpy test results on P1S1 (pipe 1, section 1) base metal orientation L.

<sup>&</sup>lt;sup>2</sup> B = specimen broken in two halves upon impact; FB = specimen unbroken upon impact but can be broken with bare fingers without using any tool (finger broken); NB = not broken (*i.e.*, cannot be broken with bare fingers).

<sup>&</sup>lt;sup>3</sup> N/A = not available (instrumented data not acquired or instrumented data analysis not reliable, particularly in the case of fully brittle tests).

Specimen ID	т (°С)	<i>к</i> (J)	<i>LE</i> (mm)	SFA (%)	B/FB/NB
D1-L3	-196	5.6	0.07	N/A	В
D1-L4	-150	8.1	0.17	N/A	FB
F1-L4	-100	7.0	0.14	23	FB
F1-L3	-75	8.7	0.15	48	В
D1-L2	-50	15.4	0.22	59	FB
F1-L2	-25	13.9	0.19	N/A	FB
D1-L1	0	10.6	0.19	55	FB
F1-L1	21	12.8	0.19	N/a	FB

Table 11 - Third-size Charpy test results on P1S2 (pipe 1, section 2) base metal orientation L.

Table 12 - Third-size Charpy test results on P2S1 (pipe 2, section 1) base metal orientation L.

Specimen ID	т (°С)	<i>КV</i> (J)	<i>LE</i> (mm)	SFA (%)	B/FB/NB
I3-L3	-196	3.3	0.03	N/A	В
G3-L4	-153	3.0	0.05	N/A	В
13-L4	-100	6.7	0.10	12	В
G3-L3	-75	6.3	0.14	30	FB
13-L2	-50	12.1	0.24	57	FB
G3-L2	-25	11.9	0.26	67	FB
13-L1	0	17.1	0.23	85	FB
G3-L1	21	9.8	0.24	100	FB

Table 13 - Third-size Charpy test results on P2S2 (pipe 2, section 2) base metal orientation L.

Specimen ID	т (°С)	<i>к</i> (J)	<i>LE</i> (mm)	SFA (%)	B/FB/NB
K1-L1	-196	5.0	0.01	N/A	В
K1-L2	-140	3.3	0.03	N/A	В
K1-L3	-100	11.0	0.21	49	FB
K1-L4	-75	7.6	0.18	31	FB
J3-L4	-50	10.2	0.15	47	FB
J3-L3	-25	11.6	0.24	76	FB
J3-L2	0	9.5	0.23	73	FB
J3-L1	21	10.5	0.22	100	FB

Specimen ID	т (°С)	<i>к</i> (J)	<i>LE</i> (mm)	SFA (%)	B/FB/NB
M18-L1	-196	0.1	0.01	N/A	В
M18-L3	-146	4.1	0.03	N/A	В
08-L2	-120	0.1	0.04	N/A	В
08-L3	-102	0.1	0.06	N/A	В
M18-L2	-75	4.3	0.04	N/A	В
M18-L4	-60	10.5	0.29	22	FB
P16-L4	-50	11.4	0.30	35	FB
08-L4	-40	15.7	0.28	25	FB
P16-L3	-25	17.4	0.47	85	NB
P16-L2	0	19.1	0.48	84	NB
P16-L1	21	20.7	0.54	100	NB
08-L1	100	19.6	0.56	100	NB

Table 14 - Third-size Charpy test results on P3S1 (pipe 3, section 1) base metal orientation L.

Table 15 - Third-size Charpy test results on P3S2 (pipe 3, section 2) base metal orientation L.

Specimen ID	т (°С)	<i>КV</i> (J)	<i>LE</i> (mm)	SFA (%)	B/FB/NB
N10-L1	-196	0.1	0.06	N/A	В
P12-L1	-150	0.9	0.1	12	В
P12-L2	-125	0.1	0.02	N/A	В
M12-L4	-90	9.5	0.18	31	FB
N10-L3	-75	N/A <sup>4</sup>	0.12	11	FB
M12-L3	-75	3.6	0.05	N/A	В
M12-L2	-50	8.4	0.25	19	FB
P12-L3	-40	8.8	0.23	27	FB
M12-L1	-25	15.7	0.34	21	NB
N10-L4	0	25.4	0.53	78	NB
N10-L2	21	23.6	0.47	100	NB
P12-L4	100	19.4	0.54	100	NB

A free copy of this report can be obtained from:

<sup>&</sup>lt;sup>4</sup> Absorbed energy value not acquired.

Specimen ID	т (°С)	<i>к</i> ∨ (J)	<i>LE</i> (mm)	SFA (%)	B/FB/NB
Q3-L1	-196	1.4	0.00	N/A	В
R9-L1	-150	0.6	0.01	N/A	В
S22-L4	-125	3.8	0.08	50	В
S22-L1	-100	5.4	0.14	24	FB
R9-L4	-85	4.8	0.18	N/A	FB
Q3-L2	-75	9.8	0.14	100	FB
Q3-L4	-65	4.2	0.21	100	FB
R9-L2	-50	13.0	0.27	100	FB
S22-L2	-25	13.3	0.20	100	FB
Q3-L3	0	11.0	0.23	N/A	FB
R9-L3	21	14.5	0.24	100	FB
S22-L3	97	13.7	0.29	100	FB

Table 16 - Third-size Charpy test results on P3S3 (pipe 3, section 3) base metal orientation L.

Absorbed energy transition curves obtained in the L orientation of the base metal for all pipes and sections are compared in Figure 24, while Table 17 collects values of DBTT and USE obtained for all conditions examined. The same values are compared in the bar charts illustrated in Figure 25 and Figure 26, respectively.

Table 17 - Ductile-to-brittle transition temperatures and upper shelf energies for base metals in L orientation, with estimated standard errors. For *DBTT*, standard error was estimated using the NIST statistical online tool "Transition Curve Fitting Tool" [X]; for *USE*, standard error was calculated based on the standard deviation of the absorbed energy values corresponding to the upper shelf of the curve.

Pipe	Section	DBTT (°C)	USE (J)
Ρ1	S1	-122.9 ± 15.7	11.0 ± 0.7
	S2	-75.0 ± 0.0012	13.2 ± 1.0
P2	S1	-77.0 ± 31.0	12.7 ± 1.6
	S2	-124.6 ± 12.6	10.1 ± 0.6
Р3	S1	-57.7 ± 2.8	19.8 ± 0.5
	S2	-42.7 ± 9.7	22.8 ± 1.8
	S3	-80.9 ± 10.4	13.1 ± 0.6



Figure 24 - Absorbed energy transition curves obtained on the base metals of the different pipes and sections in L orientation.



Figure 25 - Comparison between DBTT values calculated for the base metals of the different pipes and sections in L orientation. Note: the toughest conditions correspond to the tallest bars (P1S1 and P2S2).



Figure 26 - Comparison between USE values calculated for the base metals of the different pipes and sections in L orientation.

#### 2.10.2. Charpy test results at 0 °C for base metals in T direction

Values of absorbed energy obtained at 0 °C from instrumented Charpy tests on third-size specimens from base metals of the different pipes and sections in the transverse orientation are provided in Table 18, which also reports average *KV* values, as well as values of lateral expansion and estimated shear fracture appearance.

API 5L section 9.8 and Table 8 prescribe the minimum absorbed energy requirements for pipe body of PSL 2 pipes. For X100 pipes with outside diameter, *D*, between 762 mm and 914 mm, such as the three pipes investigated (P1, P2, and P3), the minimum required average absorbed energy,  $\overline{KV}_{min}$ , is 54 J. However, according to section 9.8.1.1, if subsize specimens are used,  $\overline{KV}_{min}$  shall be multiplied by the ratio of the subsize specimen to the full-size specimen width, W. For third-size specimens, therefore,  $\overline{KV}_{min}$  shall be divided by 3, so that:  $\overline{KV}_{min} = 54/3 = 18$  J. Moreover, according to section 9.8.1.2, absorbed energy values from each individual test,  $KV_{i}$ , must be at least equal to 75 % of  $\overline{KV}_{min}$ . For third-size specimens,  $KV_{i,1/3} \ge 14$  J (rounded to the nearest joule from 13.5 J).

The above requirements only apply to specimens in T direction and from the 90° clock position (results shown in bold in Table 18).

In Table 18, test results that do not fulfil the API 5L requirements are highlighted in red font over pink background. If they satisfy API 5L, they are highlighted in green.

Pipe	Section	Angle	Specimen	KV	KV <sub>mean</sub>	LE	<b>SFA</b> <sub>est</sub>	B/FB/NB
			id	(L)	(L)	(mm)	(%)	
			A1-T1	5.03		0.15	N/A	FB
		0°	A1-T2	6.17	7.84 ± 2.63	0.16	N/A	FB
			A1-13	9.87		0.15	N/A	FB
			A1-14	10.29		0.18	N/A	FB
			D13-11 D12 T2	6.95		0.17	85 100	FD ED
	S1	90°	B13-12	11 90	8.78 ± 2.32	0.15	100	гD СD
			B13-T3	9.38		0.15	72	FB
			C12-T1	7.25		0.14	63	FB
			C12-T2	6.69		0.15	100	FB
P1		180°	C12-T3	9.09	8.99 ± 2.82	0.15	71	FB
			C12-T4	12.94		0.17	100	FB
			D3-T1	6.17		0.16	79	FB
		0°	D3-T2	5.75	0 00 1 2 40	0.18	83	FB
		0	D3-T3	9.88	8.09 ± 2.49	0.19	61	FB
	\$2		D3-T4	10.58		0.15	62	FB
	52		F3-T1	6.96		0.15	61	В
		180°	F3-T2	9.71	9.71 ± 1.98	0.15	52	FB
		100	F3-T3	11.52		0.18	62	FB
			F3-T4	10.64		0.17	71	FB
	S1 -		G1-T1	6.40		0.12	67	В
		0°	G1-T2	7.11	8.89 ± 2.49	0.15	65	FB
			G1-T3	10.66	0.00	0.17	62	FB
			G1-T4	11.37		0.17	68	FB
			11-11	5.88		0.17	56	FB
		180°	11-12	0.03	8.13 ± 2.51	0.10	04 70	ГВ
D2			11-15	10.10		0.17	70 62	
F Z			11-14 11-T1	5 80		0.17	73	FB
		0°	11-T2	8.21	7.74 ± 1.75	0.18	68	FB
		0	J1-T3	9.21		0.19	81	В
	S2	90°	K3-T1	7.68		0.18	79	FB
			КЗ-Т2	7.54		0.19	63	FB
			КЗ-ТЗ	10.23	$9.53 \pm 2.43$	0.19	62	FB
			КЗ-Т4	12.66		0.17	74	FB
			P18-T1	7.59		0.33	100	FB
	S1	90°	P18-T2	5.62	8 31 + 7 18	0.30	100	FB
	51	50	P18-T3	9.45	0.51 ± 2.10	0.29	100	FB
			P18-T4	10.59		0.28	85	FB
			M14-T1	8.81		0.30	73	FB
	S2	90°	M14-T2	8.67	9.88 ± 1.47	0.29	100	FB
			M14-T3	10.23		0.27	85	FB
			IVI14-14	7 17		0.10	100	FB ED
			Q1-11 01-T2	1.1/		0.19	100	LD LD
Р3		0°	01-12	10.01	8.96 ± 2.5	0.19	200	FD
			01-T4	11 88		0.10	84	FR
			R11-T1	8.53		0.19	73	FB
	_		R11-T2	8.39	10 50 . 0 5	0.18	69	FB
	S3	90°	R11-T3	10.38	$10.53 \pm 3.0$	0.20	61	FB
			R11-T4	14.81		0.20	85	FB
			S24-T1	7.45		0.19	81	FB
		190%	S24-T2	8.44	0 17 + 2 22	0.21	84	FB
		190	S24-T3	8.33	9.11 ± 2.23	0.18	89	FB
			S24-T4	12.44		0.22	69	FB

Table 18 - Results of 0 °C third-size Charpy tests on base metals in T orientation. Values subject to API 5L requirements are shown in bold. Acceptable values are highlighted in green, non-acceptable values in red.

# 2.10.3. Charpy test results at 0 °C on seam weld metals and heat affected zones

Values of absorbed energy obtained at 0 °C from instrumented Charpy tests on third-size specimens extracted from seam weld metals and heat affected zones (HAZ) for the different pipes and sections are collected in Table 19, which also reports average *KV* values, as well as values of lateral expansion and estimated shear fracture appearance.

For pipe weld and HAZ tests, API 5L section 9.8.3 requires  $\overline{KV}_{min}$  = 40 J at 0 °C for pipes in grades > X80 and full-size Charpy specimens. Using the normalization procedure already described in 3.3.2, the minimum for third-size specimen becomes  $\overline{KV}_{min,1/3}$  = 13 J (rounded to the nearest joule from 13.3 J). Absorbed energy values from each individual test,  $KV_i$ , must be at least equal to 75 % of  $\overline{KV}_{min,1/3}$ . For third-size specimens,  $KV_{i,1/3} \ge 10$  J.

In Table 19, test results that do not fulfil the API 5L requirements are highlighted in red font over pink background. If they satisfy API 5L, they are highlighted in green.

Pipe	Section	Material	Specimen id	<i>кv</i> (J)	KV <sub>mean</sub> (J)	<i>LE</i> (mm)	SFA <sub>est</sub> (%)	B/FB/NB
Р1	S1	Weld	A2-W1	2.27	4.77 ± 2.17	0.09	7	В
			A2-W2	5.80		0.10	14	В
			A2-W3	6.23		0.11	7	В
		HAZ	A2-H1	6.23	6.95 ± 2.01	0.17	70	FB
			A2-H2	5.39		0.13	64	FB
			A2-H3	9.22		0.15	83	FB
	S2	Weld	D2-W1	6.07	4.43 ± 2.26	0.15	6	В
			D2-W2	5.37		0.03	18	В
			D2-W3	1.86		0.12	6	В
		HAZ	D2-H1	6.24	6.48 ± 1.92	0.17	40	FB
			D2-H2	4.69		0.14	18	FB
			D2-H3	8.51		0.13	47	FB
Ρ2	S1	Weld	G2-W1	2.98	5.15 ± 2.27	0.11	12	В
			G2-W2	4.96		0.11	17	В
			G2-W3	7.50		0.15	15	В
		HAZ	G2-H1	10.08	8.23 ± 2.28	0.15	80	FB
			G2-H2	8.93		0.14	76	FB
			G2-H3	5.68		0.13	61	FB
	S2	Weld	J2-W1	7.65	5.67 ± 2.8	0.16	16	В
			J2-W2	3.69		0.14	14	В
		HAZ	J2-H1	11.06	9 29 + 2 5	0.16	53	В
			J2-H2	7.52	5.25 ± 2.5	0.17	68	FB
	53	Weld	Q2-W1	2.97	3.63 ± 1.66	0.13	30	В
			Q2-W2	2.41		0.11	17	В
			Q2-W3	5.52		0.11	17	В
Ρ3		HAZ	Q2-H1	5.24	7.7 ± 2.15	0.15	20	В
			Q2-H2	8.66		0.17	33	В
			Q2-H3	9.21		0.17	53	В
		Weld	Q14-W1	2.78	3 35 + 0 8	0.08	20	В
			Q14-W2	3.91	5.55 ± 0.8	0.08	22	В
		HAZ	Q14-H1	5.67	9.09 ± 3.29	0.16	73	В
			Q14-H2	9.36		0.20	70	В
			Q14-H3	12.23		0.19	100	В

Table 19 – Results of third-size Charpy tests at 0 °C on seam welds and HAZ. Acceptable values according to API 5L are highlighted in green, nonacceptable values in red.

# 2.11. Charpy test results at 0 °C on girth weld metals and heat affected zones

Values of absorbed energy obtained at 0 °C from instrumented Charpy tests on third-size specimens extracted from girth weld metals and heat affected zones (HAZ) for the different pipes and sections are collected in Table 20, which also reports values of lateral expansion and estimated shear fracture appearance.

There are no absorbed energy requirements in API 5L for girth welds in PSL 2 Pipes.

Pipe	Angle	Material	Specimen ID	<i>KV</i> (J)	LE (mm)	SFA (%)	B/FB/NB
P1	90°	Weld	B2-W1	16.01	0.34	65	В
		HAZ	B2-H1	11.50	0.31	100	FB
P2	90°	Weld	H2-W1	14.84	0.33	73	FB
		HAZ	H2-H1	17.97	0.30	100	FB
Р3	0°	Weld	M17-W1	16.29	0.50	N/A	FB
		HAZ	M17-H1	23.19	0.57	100	NB
	90°	Weld	N11-W1	11.84	0.41	N/A	FB
		HAZ	N11-H1	21.73	0.58	100	NB

Table 20 - Results of third-size Charpy tests at 0 °C on girth welds and HAZ.

### 2.12. Indentation Testing

Vickers hardness indentation traces of base metal specimens are presented in Figure 27 to determine whether there is any inhomogeneity in the properties through the thickness of the pipe. A partial through-thickness nano-indentation trace is shown in Figure 28 and a nano-indentation trace along the inner surface of the pipe is shown in Figure 29. The nano-indentation traces are intended to examine whether there is any variation in material properties based on microstructural changes at the surface of the pipe.

Vickers indentation mapping was used to visualize the variation in mechanical properties over girth welds, including base metal, weld metal, and heat affected zone. Maps of the two girth welds between the 0.25" (6.35 mm) thick pipe sections are shown in Figure 30 and Figure 31.

Knoop hardness indentation traces were made across girth and seam welds to duplicate the tests presented by R. S. Ryan. Five parallel traces were made across each weld, as shown in Figure 32 (girth weld) and Figure 33 (seam weld).

All raw indentation data is provided in the project data repository (see Section 1.4).



Figure 27 – Vickers indentation trace of various base metal specimens.



Figure 28 - Nano-indentation hardness and elastic modulus originating from the inner surface of the pipe (specimen XMX RTT).



Figure 29 - Nano-indentation hardness and elastic modulus tracing the inner surface of a pipe section (specimen XMX RTT).


Vickers Indentation Hardness (HV)

Figure 30 – Vickers indentation hardness map of girth weld (EM1 ATT)





Figure 32 – Knoop hardness traces across girth weld (EM1 ATT)



Figure 33 – Knoop hardness traces across seam weld (AM2 RTT)

## 3. Discussion and Comparison

## 3.1. Chemical Analysis and Pipe Identification

To assist in determining the origin of the received pipe the chemical analysis data was compared to those in the 37 NG-18 reports provided by PRCI. Several experimental steels were detailed throughout the NG-18 reports, including information on chemistry, heat treatment, wall thickness, and summary mechanical properties. Unfortunately, there were no steels that matched the chemistry or summary mechanical properties of the vintage Columbia Gas X100 pipe. In some cases, full Charpy impact energy curves were provided in the NG-18 reports, but comparison is not possible without verification that the received vintage Columbia Gas X100 pipe is the same material listed in the tests.

Further complicating the identification of the pipe is the variation in the chemistry of the seven individual pipe sections. It is understood that sections P3S1 and P3S2 (0.45"/11.43 mm wall thickness) is a specimen of X60 based on the Columbia Gas historical documents. However, the variation in chemical data from optical emission spectroscopy seems to suggest that pipe sections P1S1, P1S2, P2S1, P2S2, and P3S3 may in fact all be different experimental materials.

## **3.2.** Microstructure Analysis

The microstructure of base metal, heat-affected zones (HAZ), and welds were analyzed with optical microscopy, SEM backscatter imaging, and EBSD to determine general microstructural morphology, grain size, grain morphology, relative misorientation, and crystallographic alignment. The welds and HAZ contain a mix of martensite, ferrite, and bainite, as indicated by optical and BSE imaging, which is consistent with previous work on X100 pipeline steel [27]. Given the fine size scale of bainite, which is a mixture of ferrite and cementite, and the uncertainty in using EBSD to differentiate the body-centered cubic (BCC) ferrite phase from tetragonal martensite phase, it is common practice to index the microstructure using only the ferrite phase during EBSD measurements and use specific microstructural characteristics to discern between the various features [45], [46]. The base metal of API X100 steel is composed of mostly polygonal ferrite and some acicular ferrite morphologies, but dynamic

recrystallization can occur, depending on the rolling temperature employed during processing [28]. While rolling textures are an effective way of steering properties for a given application, sub-grain structures (dislocation arrays) and grain boundary character/density are economically viable for generating higher strengths [30].

Since the girth welds resulted in greater Charpy strength and higher variability in Knoop hardness and grain size (via optical and BSE imaging) as compared to seam welds, large-area EBSD scans were employed from weld to HAZ to base metal. Previous work on X100 steel welds showed the importance of minimizing coarsened martensite-austenite constituents in the heat affected zone as these features led to an increase in microcracking and cleavage [34]. Further, multiple HAZs have been reported in welded X100 pipeline samples, characterized by a transition in grain size, intragranular misorientation, and grain boundary angle [47]. Grain-size based nomenclature is most commonly employed to describe the differences, such as finegrained HAZ (FG-HAZ) and coarse-grained HAZ (CG-HAZ). In the current work, the trend of multiple HAZs is also observed. The HAZ closest to the weld is characterized by an extremely refined grain structure (approximately 3 um diameter globular ferrite), higher kernel misorientation, and increased grain orientation deviation as compared to the weld. The columnar parent austenite grains seen in the weld were longer than 1 mm in many cases and is influenced by welding speed and interpass temperature [48]. The HAZ region closest to the base metal also showed evidence of grain refinement compared to the base metal (approximately 7 um grain diameter) but was not as drastic of a difference as depicted in the HAZ closest to the weld. Such differences explain the trends in Knoop hardness where a nearly 30% increase in hardness at the weld-HAZ boundary whereas a drastic decrease in hardness is observed near the base metal-HAZ boundary. It is also noteworthy to mention that tensile specimens containing a girth weld tended to fracture at the HAZ. This is likely due to drastic changes in intragranular misorientation. The average GROD value in degrees of orientation deviation respectively changed from  $\sim 5^{\circ}$  in the weld to  $\sim 4^{\circ}$  in the HAZ near the weld to  $\sim 2^{\circ}$  in the HAZ near the base metal and back to approximately  $\sim 4^{\circ}$  in the base metal.

With respect to the base metal, interesting features were observed near the surface of the pipe (outer diameter) in a refined grain structure exists and is likely caused by processing routines

A free copy of this report can be obtained from: https://doi.org/10.6028/NIST.IR.8535 and environmental effects over time. The refined portion tends to have more randomly oriented grains, lower grain orientation deviation and a smaller grain size. When all three orthogonal planes were analyzed, the trend remained. However, given the small volume fraction this region encompasses in full-scale tensile specimens, structure-property correlations were not observed. Rather, the typical bainite-ferrite microstructure observed with equiaxed parent austenite grains indicates a nearly complete recrystallization of grain structure in the EM3 pipe section.

#### **3.3.** Tensile Testing

At the time that this X100 pipe was produced, specifications for X100 line pipe did not exist. However, ASTM A370 and ASTM E8 standards did exist and were referenced in the API 5L specification. This is important because some differences in the data between the tests conducted at the time the pipe was produced and now, may be attributed to the differences in test method, specimen preparation, measurement methods or in the analysis. API 5L, ASTM A370 and ASTM E8 all existed at the time the X100 was produced and tested. Without knowing the exact time of production, each of the standards were all updated and published in 1966. For reference to potential changes, the 1966 versions will be compared to current versions.

The current version of API 5L significantly differs from the 1966 version, reflecting advancements in technology and industry standards. The 1966 version is limited in grade specification, while the current version includes higher strength grades like X70, X80, X100, and X120, addressing the need for stronger materials. The introduction of Product Specification Levels (PSL1 and PSL2) in the current version brings stricter requirements for chemical composition, mechanical properties, and testing. Modern testing methods, such as nondestructive testing and Charpy V-Notch impact testing, ensure higher reliability. Additionally, the current version specifies detailed manufacturing processes and enforces stricter controls over chemical composition.

The ASTM A370 standard has seen substantial improvements since its 1966 version, mostly reflecting advancements in measurement technology and test equipment and testing method. The current standard incorporates modern testing equipment and methods, enhancing the

accuracy of measurement requirement. It also provides more detailed testing procedures, improved safety guidelines, and stricter reporting requirements. It integrates advancements in data acquisition and analysis, addressing previous revisions and errata to enhance clarity and usability.

The current version of ASTM E8 has significantly evolved from the 1966 version, incorporating advanced test control methods (Stress Rate, Strain Rate, Crosshead Displacement), and improved gripping technologies for better accuracy. It mandates high-accuracy extensometers for precise strain measurement and provides detailed guidelines for specimen preparation.

There is very little information known about the testing performed on this X100 prior to being placed into service. However, full thickness test specimens had the same specimen geometry requirements as are relevant to the testing reported herein. Differences in test machines and gripping methods are known to affect the test data but it would be difficult to determine an uncertainty associated with those. Furthermore, flattening procedures have not been standardized even for current testing, adding yet another uncertainty element that is difficult to enumerate. Lastly, advances in measurement technologies, to include high-precision extensometers, digital data acquisition systems, and computerized data analysis have the largest potential for significant differences in test data.

Examining the tensile data in Table 9, most of the pipe sections met the current tensile requirements for API 5L X100Q. There are notable sections that failed to meet the minimum requirements.

The average value from section P2S2-90 included three test specimens that were all tested on the same day with the same setup and instrumentation. All three specimens exhibited anomalous strain data evidenced by very low modulus values. If low modulus values are the result of a test or measurement error, it is reasonable to suggest that if the correct modulus values (from suspect strain values) were calculated, then the yield strengths (0.5 %) would have shifted and therefore would have met the minimum required yield strength. The 0.2 % offset yield strength is less sensitive to errors or changes in modulus. Even with anomalous strain values, one of the three specimens would have passed with a yield strength (0.5 %) of 101.9 ksi,

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it was the other two specimens that brought the average below specification at 95.6 ksi and 96.4 ksi respectively. The anomaly is still being investigated and those data sets will not be included in the data repository until the data can be further validated. All other tensile data associated with those three specimens are consistent with tests from other sections. Additionally, those specimens were from the 90 ° circumferential orientation, which is required by API 5L, but specimens tested from P2S2-180 all passed the minimum specification. It is unlikely that the material at the 90 ° position was that significantly different from the 180 ° position, especially when all other 90 ° and 180 ° data are effectively equal within the standard deviation from other pipe sections.

The two sections, P3S1 and P3S2, were made from an X60 grade steel, while they are included in the data, they are not compared to X100 in anyway. These sections have a wall thickness of 0.45 in (11.4 mm), to carry the design pressure of 1,000 psi (6.9 MPa). See Appendix A for additional details about hydro-testing and some material information.

#### 3.4. Instrumented Charpy Testing

#### 3.4.1. Base metals, orientation L (transition curves)

API 5L does not contain provisions or requirements pertaining to the base metal (pipe body) of PSL 2 pipes in longitudinal orientation. The comparison between absorbed energy transition curves, illustrated in Figure 24, shows that P3S1 and P3S2 exhibited the highest upper shelf energies (these are X60), while the lowest<sup>5</sup> *DBTT* values were yielded by P2S2 and P1S1. This is confirmed by the bar charts in Figure 25 (*USE*) and Figure 26 (*DBTT*). It's interesting to note the lowest value of *USE* corresponds to the lowest value of *DBTT* (P2S2). It's also noteworthy that several investigated conditions (P1S2, P2S1, P2S2) displayed a relatively high lower shelf energy, even at liquid nitrogen temperature (-196 °C). A fair amount of data scatter can be observed for all the investigated conditions.

<sup>&</sup>lt;sup>5</sup> As a reminder, a decrease of *DBTT* corresponds to an increase in toughness.

With respect to the Charpy data presented by R. S. Ryan in [1], as already mentioned, we ignore if they were obtained in L or T orientation. The upper shelf energy in Figure 2 (left side) is approximately 33 J, which is much higher than any *USE* value yielded by the pipes/sections considered in this investigation (10. 1 J to 22.8 J, Table 18 and Figure 25). We cannot exclude, however, that the results in [1] were obtained from larger subsize specimens, for example halfsize (B = 5 mm, W = 10 mm). In terms of DBTT, the values from [1], which correspond to -33 °C for absorbed energy and -43 °C for shear fracture appearance, are higher than most of the *DBTT* values recorded in this investigation (-124.6 °C to -42.7 °C, Table 18 and Figure 24).

## 3.4.2. Base metals, orientation T (0 °C)

All pipes and sections were characterized in different clock orientations by performing thirdsize instrumented Charpy tests at 0 °C. For specimens extracted in the 90° clock position, API 5L prescribes minimum values for the average absorbed energy, as well as for individual *KV* values. These requirements, normalized by the ratio of subsize/full-size specimen widths, correspond to  $\overline{KV}_{1/3} \ge 18$  J and  $KV_{i,1/3} \ge 14$  J for X100 line pipe.

As shown in Table 18, none of the average values of absorbed energy for specimens extracted in the 90° clock position satisfied the API 5L requirement. As for individual *KV* values, only one out of 20 X100 specimens tested in the 90° clock position met the API 5L requirement

(specimen R11-T4 from P2S2, *KV* = 14.81 J).

## 3.4.3. Seam weld metals and HAZ (0 °C)

The weld metals and heat affected zones of the seam welds of the investigated pipes were characterized by testing third-size Charpy specimens at 0 °C. For seam welds, API 5L prescribes  $\overline{KV}_{1/3} \ge 13$  J and  $KV_{i,1/3} \ge 10$  J (after normalization based on subsize specimen widths). As can be seen in Table 19, only one of the 12 average absorbed energy values meet the API 5L requirements. As for individual energy values, only 6 out of 33 tested specimens, all from HAZ, absorbed more than the API 5L minimum. None of the weld metal results were acceptable.

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## 3.4.4. Girth weld metals and HAZ (0 °C)

The weld metals and heat affected zones of the girth welds of the investigated pipes were characterized by testing third-size Charpy specimens at 0 °C. There are no requirements for girth welds of PSL 2 pipes in API 5L.

The comparison between Table 20 (girth welds) and Table 19 (seam welds) shows that, for the three investigated pipes, girth welds are significantly tougher than seam welds. Specifically, the average absorbed energies were 4.50 J (weld metals) and 7.88 J (HAZ) for seam welds, and 14.75 J (weld metals) and 18.60 J (HAZ) for girth welds.

## **3.5.** Indentation Testing

#### 3.5.1. Base Metal

Micro-indentation traces across through-thickness sections of base metal indicate that there is no significant inhomogeneous degradation of the pipe due to the long-term exposure to natural gas and/or corrosive natural elements. The full through-thickness hardness for the 6.35 mm thick X100 pipe in sections P1S1, P1S2, and P2S1, presented in Figure 27, illustrates a hardness of approximately 300 HV with no significant variation through the thickness of the pipe. Note that the steep decreases at the end of the indentation hardness traces are caused by indentations in the phenolic resin mounting media at the end of the indent trace.

The through-thickness Vickers hardness of the thicker 11.43 mm pipe (assumed to be vintage X60 pipe per Columba Gas documents) shows a much lower hardness of about 200 HV. There is no historical data to compare to the thicker vintage X60 steel.

To supplement the through-thickness micro-indentation traces, nano-indentation hardness (Figure 28) was performed to investigate any material property changes related to the significantly smaller grain size at the inner surface of the vintage X100 steel. Nano-indentation hardness can be performed with much higher spatial resolution than micro-indentation hardness without causing interaction of the indentation deformation volumes. Instrumented nano-indentation also allows for calculation of the localized elastic modulus. With the higher spatial resolution of nano-indentation there appears to be a reduction in nanoindentation hardness at the inner surface (about 3.5 GPa) compared to the bulk base metal (about 4 GPa). This reduction can be seen in the through-thickness nano-indentation trace (Figure 27) and confirmed in the nano-indentation traverse along the inner pipe diameter (Figure 28).

#### 3.5.1.1. Seam Welds and Girth Welds

Knoop hardness indentation traverses were made across both girth and seam welds to duplicate the data presented by R. S. Ryan (Figure 1).

Hardness traverses across a seam weld (Figure 33) illustrate almost no change in indentation hardness through base metal, heat affected zone, and weld metal. This seems reasonable as R.S. Ryan indicates that the seam welds were performed during pipe fabrication and the welded pipe was heat treated following welding. Though there is not variation across the weld, the base metal hardness is approximately 325 HK, which is consistent with the original data from R.S. Ryan. Based on the base metal adjacent to the seam weld(s), there was no significant degradation in hardness after long term exposure to natural gas and/or environmental elements.

The flat hardness curve across the seam weld corresponds well with the homogenous microstructure and grain size of the seam welds. The post-weld heat treatment likely homogenized the weld microstructure and subsequently the mechanical properties.

Unlike the seam welds, the pipe girth welds were performed on site during pipe installation and were not heat treated. Based on the seam weld traverse, the original R. S. Ryan data (Figure 1) appears to be from a non-heat-treated girth weld.

Vickers indentations were used to map the hardness across the entire cross section of girth welds. All tested girth welds (examples in Figure 30 and Figure 31) show an increase in hardness in the heat affected zone, especially adjacent to the weld cap. This is in addition to a reduction in hardness (relative to the base metal) directly between the heat affected zone and the base

metal. This is partially consistent with the data from the original publication, though R.S. Ryan reported that the heat affected zone was not harder than the weld metal.

Five Knoop indentation traverses were performed across girth welds to duplicate the measurement presented by R.S. Ryan. All girth weld hardness traverses (representative data in Figure 32) had some similarities with the R.S. Ryan, specifically the base metal and weld metal had approximately the same hardness and there is a notable reduction in hardness when traversing from base metal through the heat affected zone. In the modern measurements the heat affected zone is significantly harder than the base metal or weld metal. The hardness profile of Traverse 5 in Figure 32, which is near the center of the pipe thickness, is most like the R.S. Ryan hardness traverse.

When comparing the weld microstructure and the hardness trace(s), the increased hardness in the heat affected zone is correlated with the reduced grain size in the HAZ of the girth welds as illustrated in the SEM backscattered imaging in Figure 33.

Like other data presented in this report, the weld hardness traces are difficult to directly compare to the original R. S. Ryan data. Aside from the fact that the indentation load is not reported by R.S. Ryan, it is clear from Figure 32 that the exact position of the hardness traverse within the weld cross section has a significant impact on the hardness profile.

Lastly, an experimental atomic force microscopy (AFM) method called contact resonance was attempted to determine highly localized variation in the material elastic properties. Contact resonance AFM measures the mechanical properties by measuring the change in cantilever resonance frequency as the AFM tip interacts with the specimen. Unfortunately, there was not enough variation in local elastic properties to allow detection using CR-AFM.

#### 4. Conclusions

Microstructural analysis and chemical analysis, including optical emission spectroscopy, optical imaging, backscatter electron imaging, and electron backscatter diffraction was performed on all provided pipe sections. The main conclusions of the microstructural analysis are the following.

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- 1. Chemical composition was inconsistent between the seven distinct pipe sections. It is possible that each pipe section is a different experimental material.
- 2. All base metal pipe sections displayed a bainite-ferrite microstructure.
- 3. Reduced grain size was observed at the inner and outer surfaces of pipe in cross sectional microstructural imaging.
- 4. Annealed seam welds displayed little difference in microstructure between the base metal, weld, and heat affected zone.
- 5. Unannealed girth welds illustrated a significant reduction in grain size within the heat affected zone.

Circumferential tension tests on full thickness flattened strap tensile specimens were performed on pipe body base metal and seam welds. The main remarks emerging from the results obtained are the following.

- Test methods used to evaluate the steel line pipe prior to installation in the 1960's are similar to current standards in their method, but advances of instrumentation and data acquisition make direct comparisons challenging with uncertainties from the 1960's being the largest factor.
- 2. The lack of construction and test data prior to putting the pipes into service make it impossible to determine if the pipes experienced any time-history effects on the tensile properties.
- 3. The vintage X100 steel line pipe would satisfy the current API 5L minimum tensile requirements for X100Q steel line pipe, with minor notable caveats related to potential testing errors associated with small sample sizes.
- 4. Circumferential tensile properties of this vintage X100 steel line pipe are in alignment with the tensile properties and performance of modern X100 steel.

Instrumented Charpy tests on third-size specimens (B = 3.3 mm, W = 10 mm) were performed on base metals in L and T orientations, as well as on weld metals and heat affected zones of seam and girth welds. The main observations emerging from the results obtained are the following.

- Based on absorbed energy transition curves obtained for longitudinal specimens, the least tough pipe sections are P1S1 and P2S2 (highest *DBTT* and lowest *USE*). The toughest pipes/sections are P3S1 and P3S2 (lowest *DBTT* and highest *USE*), noting that those are an X60 grade pipe.
- 2. For the base metals in T direction, tests were performed at 0 °C. Considering the API 5L requirements on absorbed energy for specimens extracted in the 90° clock position, none of the pipes satisfies the requirement based on the average absorbed energy,

while only 1 out of 20 specimens tested exceeds the minimum *KV* required (14 J) by the API specification.

- 3. For the seam weld metals and HAZ tested at 0 °C, the API 5L requirement on the mean absorbed energy was only met by one HAZ sample. The requirement on the minimum *KV* of an individual test was satisfied by just 6 specimens out of 33 tested, all from HAZ material.
- 4. There are no requirements in API 5L for the girth weld metals and HAZ. The results obtained at 0 °C consistently show higher impact toughness than seam welds.

Micro-indentation (Vickers and Knoop) and instrumented nano-indentation was performed on base metal, seam welds, and girth welds to duplicate data in the original R.S. Ryan paper and determine if any inhomogeneous degradation was present in the steels. The main conclusions of the indentation analysis are the following.

- 1. Knoop hardness made on base metal specimens showed no significant variation in hardness through the thickness of any pipe section.
- 2. Vickers hardness maps and Knoop hardness traces across girth welds showed an increase in hardness across the heat affected zone. This finding differs from the original results presented by R.S. Ryan, but it is evident that the location of the hardness trace within the girth weld cross section will significantly impact the hardness profile.
- 3. Knoop hardness traces across seam welds showed no significant variation in hardness between base metal, heat affected zone, and weld metal.
- 4. Nano-indentation illustrated a minor decrease in the indentation hardness near the internal pipe surface, but no significant change in elastic modulus.

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ray spectroscopy," *Ultramicroscopy*, vol. 147, pp. 114–132, Dec. 2014, doi: 10.1016/j.ultramic.2014.07.005.

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## Appendix A. Historical Pipeline Documents

Classified By(Initials)
Retention Period
Date Received
(Initials) Reference

Mr. C. P. Brisley Mr. Leo J. Payne 36" X-100. .250 Wall Experimental Line

Destruction Date\_

Subject

On November 25, 1964, 1193' of 36", X-100, .250 Well experimental pipe was laid east of Petersburg Valve parallel to our 26" Line WB Loop. This line was subjected to a hydrostatic test at 1183.5% psi. for a period of 14 hours. On November 30, 1964 this line was loaded through a 2" connection to 26" Line WB Loop and will float with line pressure.

A copy of the attached charts and Pipelines and Muine Test Data were mailed to Logan Wallingford at Columbus, Ohilo.

Leo J. Payne

Attachments

ces Logan Wallingford

W. P. Diehl, Jr.

## PIPELINES AND MAINS TEST DATA

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NOTE: If the line falls into more than one Construction Type (excepting road, railroad crossings, fabricated assemblies, spans, .) complete separate sheet for each type.







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# VISITORS TO ATLANTIC SEABOARD CORPORATION'S 36" X-100 EXPERIMENTAL LINE

#### NAME

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#### COMPANY

Sy Orlofsky Robert S. Ryan R. C. Wolfe Edward Dzubak Donald R. Feltz Donald A. Benjamin Daniel J. Coffee D. D. Defibaugh J. Norman Bossert Arthur L. Towne T. T. Greene Glenn Tollerene George Eichenberger Wade Gwinn Boyd Shaw Stanley Harward W. W. Ferrell Ira Good C. P. Brisley Jack G. Brown N. A. Rupe J. Frank Dickerson Camden Garrett Donald C. White R. P. Ballard

Columbia Gas System Service Corporation Manufacturers Light and Heat Company Columbia Gulf

Columbia Gulf

Ohio Fuel Gas Company Ohio Fuel Gas Company Ohio Fuel Gas Company Ohio Fuel Gas Company United Fuel Gas Company

NAME Robert L. Morris Bill E. Sebok Jon O. Loker Byron E. Ashley W. Lynn Dolly Jimmy E. Sligh Forrest H. Smith Leo J. Payne H. C. Arthur R. E. Lynn William H. Isner P. O. Hamer H. C. Mefford, Jr. E. F. Hepler P. J. Smith Bob Collett Lloyd Ulrich H. W. Morris G. G. Gum W. J. Fridley C. G. Simmons Kenneth Wright M. J. Walton F. C. Gum L. D. Goad Frank Pangle

#### COMPANY

United Fuel Gas Company United Fuel Gas Company United Fuel Gas Company United Fuel Gas Company United Fuel Gas Company Kentucky Gas Transmission Corp. Kentucky Gas Transmission Corp. Atlantic Seaboard Corporation Atlantic Seaboard Corporation

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## NAME

John Hinkle Austin Strawderman Marvin Sager E. D. Kesner John Hardy L. L. Miller, Jr. C. F. Ritenour Richard Ambrose Snowden Alt Bernie Rome Hal Gould Gordon Buck Chuck Wald Harry Burdett Earl Tice Paul Dion Kelsey Jones David Gerould Tom Morgan Ben Montgomery Cliff Hunt Roger Jones Jaye Gamble Bill Marner George Palmer Bill Heineman Robert Cunningham

#### <u>COMPANY</u>

Atlantic Seaboard Corporation American Machine Foundry Thomas Contractors Thomas Contractors Thomas Contractors Crose-Perrault Crose-Perrault Crose-Perrault Crose-Perrault U. S. Steel U. S. Steel Linde Hobart United Gas Company United Gas Company

- 3 -

T. S. Tilford William Hitchcock Art Bradfield Sam Nettles Carl Winters Dale Smith Bill Graham Ike Goodwin Joe Chapman E. G. Summers Herb Wilson George Campbell H. A. Sosnin Robert Scarborough Lou Sache Lou Mendonsa Fred Cornelius Ray Beirne Ron Prehoda

United Gas Company Texas Eastern Gas Company Southern California Gas Company Consolidated X-Ray Wilson Welding Supply Washington Gas & Light Washington Gas & Light Washington Gas & Light Crutcher-Rolf-Cummins General Pipeline Construction, Inc. General Pipeline Construction, Inc. Helicopter Patrol, Inc. Piping Consultant Federal Power Commission Federal Power Commission

# WORKERS ON ATLANTIC SEABOARD CORPORATION'S 36" X-100 EXPERIMENTAL LINE

#### NAME COMPANY Logan Wallingford Columbia Gas System Service Corporation Richard B. Gwin Columbia Gas System Service Corporation Bruce L. Hutt Columbia Gas System Service Corporation Claude W. Churchheus Columbia Gas System Service Corporation Marion Bailey Kentucky Gas Transmission Paul B. Poliskey United Fuel Gas Company Roger L. Young United Fuel Gas Company Ross V. Carper United Fuel Gas Company Atlantic Seaboard Corporation W. P. Diehl, Jr. T. E. Dean Atlantic Seaboard Corporation Henry A. Dean Atlantic Seaboard Corporation B. C. Richard Atlantic Seaboard Corporation Merle Foudray Pipeline Maintenance & Construction

COLUMBIA GAS SYSTEM

FORM CS 6-85 CSD

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FACILITY FAILURE REPORT

(CONFIDENTIAL DATA)

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COLUMBIA GAS SYSTEM

FACILITY FAILURE REPORT

(CONFIDENTIAL DATA)

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TYPE OF FACILITY THAT FAILED:		DATE OF FAILUR		COUNTY	STATE
PIPE, VALVE, POWER ROD, REGULATOR, FITTING,ETC.)		6-8-67	OF FAILURE	Grank	W. Va.
AILURE ROAD BUSIN CCURRED AT CROSSING DISTRI ESCRIBE FAILURE INCLUDING ANY PECULIARITIES OR TTACH PHOTOGRAPHS OR DRAWINGS, IF POSSIBLE, II	ESS RESIDENT ICT AREA DEFECTS IN FAILED PAR NCLUDE SKETCH OF FAIL	IAL	OTHER POS EVIDENCE OF PRIO T. ATTACH ADDITIO	R DAMAGE, PREVIOU	IS REPAIRS, ETC. CESSARY.
Describe failure. Failure occurred at to the longitudinal sear a estern direction until it hit cone of the weld shearing about onally toward the top of the ailure. This pipe was coated in the area of failure. OME TYPES OF FRACTURES HAVE CHARACTERISTIC " HAT POINT TO THE FRACTURE ORIGIN. CAN ORIGIN OCATION OF	at the 9 o'clo a girth field t 90 percent roa the origin pipe. Approx with coal tan "CHEVRON" OR "HERRING OF FRACTURE BE LOCA"	bek positi the seam weld whe of the wel for abou instely 9 cenarel a BONE" MARKS FED	on looking for appro re it ran a d. The fai t 3 feet a feet of pi nd there w	East. Fail <u>mately 4</u> : around the H Llure moved id then trai pe was effected is no evider PES	iure was adjac feet in a heat offected along the iled off dia- rted by the rce of corrosi
RACTURE ORIGIN					
	CAUSE C	F FAILURE			
AS THE CAUSE OF FAILURE EFINITELY ESTABLISHED YES	NO .	IF NOT, WERE A	NY POSSIBLE VERED	YES	NO NO
a harden de to de her in realiser d'en realiser here entre recen	an a	STATUS STATUS			
HAT CAN BE DONE TO PREVENT FUTURE FAILURE	OF THIS TYPE				
4					H
	CIRCUM	STANCES			
NTERNAL PRESSURE AT OINT OF FAILURE 1470 F	IF MECH. EQUIP., L SIG AT TIME OF FAILUR	OAD FACTOR E			
ABOVE DELOW	IF BELOW GROUND, DEPTH OF FILL	IN'S A	EMPERATURE OF P	ART	AMBIENT °F
OCATION GROUND GROUND		F	AILURE		
AILURE WAS		G O D	CCURRED URING		TEST
AILURE WAS LEAK BLOWD	OPERATIN MECHANIS OTHER IMPORTANT CIR	G b M D CUMSTANCES	CCURRED URING		TEST

									67	
				HIS	TORY					
DATE INSTALLED	WAS THE FACIL PROOF TESTED	ITY AFTER		<b></b>	TYPE OF	HYDRO-				
Nov. 1964		MININ	YES	L NO	TIME HELD	STATIC	GAS			
TEST PRESSURES	1225	PSIG	1183.5	PSIG		A.M.	P M	OPERATING	800	PSIG
LATER TEST	Т	EST	MAXIMUM	1014	MINI	MUM	1.101.	TIME HELD	000	
IN YEAR	P	RESSURES			PSIG	1	PSIG	ļ A	м. М.,	P. M
FACILITY WAS BUILT	IN ACCORDA ASA B.31.1.8	NCE WITH 3-1955		PRIOR T 1955	0		CODE			
DESIGN		CONSTR	UCTION TYPE	OR DIVISIO	N			12 I.		÷.
PRESSURE REMARKS OR OTH	1000 P	SIG CLASS	ACCORDING	TO CODE	Typ	e A				
	10 10 10 10 10 10 10 10 10 10 10 10 10 1									
			5 s					1	i e di un Li	<u> </u>
			SPEC	IFICATIO	NS OF M	ATERIAL				
(a)	SPECIFICATIONS	an a	1		MANUFA	CTURER	NON	1INAL O. D.	WALL THICKNE	SS
	(A. P. I., A. S. T. M., ET	rc.) Al			EP'S TEST	. Steel		36 IN'	s .250	IN.
	PRESSURE	mam	PSIG		ALK 3 1231		SPE	CIFIED MINIMUM	100.000	DCI
	ТҮРЕ								1009000	F3I
	SEAN	MLESS		1	RESISTAN	ICE WELDED		L FLA	SH WELDED	
PIPE	DOU	BLE SUBMER	GED ARC WEL	DED [	SINGLE S	UBMERGED AF	C WELDED		WELDED	
		T WELDED		21.	EXPANDE	D .			IEXPANDED	
					1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1. 1					
	TYPE		1.1							
					NAME	OF MANUFAC	URER			
FABRICATED	DESCRIPTION OR S	PECIFICATION		JFACTURED						
OR										
ASSEMBLY										
	MATERIAL									
	OTHER DATA		<i>1</i> 1			n Maria		e 2	100 C	
	DESCRIPTION		e . La contractione de la contractione		A	ç1.4				
	DESCRIPTION									
OPERATINO										
MECHANISM										
OR	NAME OF MANUFAC	JURER				MATERIA	AL.			
EQUIPMENT	OTHER DATA									
	,			- n -						-
DATE OF REPORT			PREPA	RED 7	1					
	June 16, 196	7	BY	No	ngla	. 1. ;	file	m		
de fault		and the second second second		Comment	1	11				

### OFFICE MEMORANDUM

CHARLESTON, W. VA.

TO Mr. L. J. Payne

FORM U 216 CSD

DATE July 16, 1965

FROM Donald C. White

SUBJECT Special 36" x 6" Weldolet Fittings for Line WB-5

Attached is a copy of a drawing received from Bonney Forge, Inc. The drawing indicates the dimensions of the  $36'' \times 6''$  Weldolet fitting, ordered under P. 0. #9198, to be used in construction of Line WB-5.

The required weld contour has been sketched on the drawing. A note has been added stating that mechanical stress relief is required between each weld pass.

1 lon

Donald C. White

DCW:jrs

Attachment

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Destruction Date \_

Retention Period
Date Received
Verified By
(Initials)
June 22, 1967
Reference

Mr. C. P. Brisley

CSD

Mr. J. Frank Dickerson

Hydrostatic Test - 36" Line WB-5

Atlantic Seaboard personnel have completed the hydrostatic testing of 36" Line WB-5 from Route 220 to the Moorefield Valve Setting. This is the section of 36" laid by Carl Smith in 1965 and includes the X-100 Experimental Pipe laid in 1964.

Desting File

Due to elevation differences it was necessary to divide this portion of line into six test sections.

Attached copies of Pressure and Temperature Charts reflect the 24-Hour Test conditions for each section. In addition, a pressure sheet listing the deadweight readings and other pertinent test information is included.

The X-60, .438 Wall Pipe in Sections 1, 2, 4, 5, and 6 has a specified minimum yield of 1460 Psi. These sections were subjected to a maximum test pressure equal to 110% of SMYS or 1606 Psi. Section #3, which contained the 1185' of Experimental Pipe, was subjected to a maximum test pressure equal to 110% of the SMYS for X-100, .250 Wall Pipe or 1529 Psi. The minimum test pressure for all sections was 1.25 of the design pressure of 1000 Psi or 1250 Psi.

The original test sheets will remain on file at Dranesville.

There were no failures on the X-60, .438 wall pipe however two seam failures were encountered on the X-100, .250 Wall Experimental Pipe. Facility Failure Reports will follow under separate cover, Pipe Line and Mains Test Data Sheets for the 51,936 feet of X-60, .438 Wall and the 1185' of X-60, .250 Wall are forwarded for your disposition.

As a result of this test, 36" Line WB-5 from Route 220 to the Moorefield Valve is qualified for operation at 1000 Psi.

> J. Frank Dickerson Staff Engineer

cc: R. E. Lynn Staf Attachments: Pressure Charts (6) Temperature Charts (6) Pressure Sheets (6) Pipe Line & Mains Test Data Sheets (2) Test Section No. 6 - 36" Line W8-5 5,240 Ft. of 36", X-60, .438" Wall Dead Weight Readings

Time	Pressure	<u>Remarks</u>
June 2, 1967		
6:25 P.M.	1470	
6:35	1607	36" Ball Valve at Moorefleld Gate Setting Leaking
6:45	1600	3
6:55	1596	
7:05	1595	
7:15	1593	
7:25	1593	
7:35	1592	Repressure
7:40	1607	
7:50	1607	
8:00	1605	
8:10	1605	
8:20	1604	
8:30	1602	
8:40	1601	
8:50	1601	
9:00	1601	
June 3, 1967		
8:00 A.M.	1588	
8:30	1588	
9:00	1588	
9:30	1588	
10:00	1588	
10:30	1588	
11:00	1588	
11:30	1588	
12:00	1579	
12:10	1579	
12:20	1579	
12:30	1579	
12:40	1579	
12:50	1579	
1:00 P.M.	1579	
1:30	1579	
2:00	1579	Repressure
2:10	1609	·
2:20	1609	•
2:30	1609	
2:40	1608	
2:50	1608	
3:00	1608	
3:10	1608	
3130	1607	
4:00	1607	
4:30	1606	
5:00	1606	
5:30	1606	
6:00	1606	
6130	1606	Test Off

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Test Section No. 4 -- 36" Line WB-5 7,000 Ft. of 36" Pipe, X-60, .438" Wall Dead Weight Readings:

<u>, Time</u>	•	<u>&amp;Pressure</u>
June 4, 1967		
9:45	A.M.	1070
10:00		1255
10:10		1251
10:20		1250
10:30		1249
10:40		1248
10:50		1247
11:00		1247
11:10		1246
11:20		1246
11:30		1246
11:40		1246
11:县0		1246
12:00		1245
12:30		1245
. 1:00	P.M.	1245
1:30		1244
2:00		1244
2:30		1243
3: <b>4</b> 0		1243
3:30		1243
3:55		1243
4:30		1243
5:00		1242
6:00		1242
7:00		1242
8:00		1242
June 5, 1967		
8:00	А.М.	1240
9100		1240
10:00		1242

<u>Remarks</u>

Start Pressuring

Test On

## Test Off

Test Section No. 1 --36" Line WB-5 7,000' of 36" Pipe, X-60, .438" Wall Dead-weight readings:

<u>enit</u>	Pressure	<u>Remarks</u>
June 6, 1967		
4128 pm	505	Start Pressuring
6:07	1606	Test on
6:17	1600	Small leak in buil plug, repaired (time 6:17 to 6:23)
6:27	1597	
6:37	1596	
6:47	1595	
6:57	1594	
7:07	1593	
7:30	1591	
8:00	1590	
June \$7 1967		
8:00 am	1582	1317# = 1582# because of difference
Moved to upp	er manifold 🗕	In elevation
9:15	1317	
10:00	1317	
11:00	1317	
12:00	1317	
1:00 P.M.	1317	
2:00	1317	
3:00	1317	
4:00	1317	
5:00	1317	
6:07	1317	Test Off

Test Section No. 2 - 36" Line WB-5 7,000' of 36" Pipe, X-60, .438" Wall Dead-weight readings:

Time	Pressure
June 7, 1967	
9:26 A.M.	1337
9:40	1532
10:12	1529
10:20	1605
10:30	1601
10:40	1600
10:50	1599
11:00	1599
11:10	1599
11:20	1599
11:30	1599_
12:00	1597
1:00 P.M.	1597 🖵
2:00	1595 /
3:00	1594 (
4:00	1593
5:00	1593
6:00	1593
June 8, 1967	
8100 A.M.	1593
9:00	1593
10:00	1593
10:20	1593

Remarks

Start Pressuring Out of Water - Adjusted recording gage with dead weight Start Pressuring Test On

Slight leak in 2" Valve

Test Off

Test Section No. 3 - 36" Line V/B-5

1,185 ft. of 36" Plpe, X-100, .250" Wall 10,015 ft. of 36" Plpe, X-60, .438" Wall

. .

Dead-weight readings:

<u></u>	<u>Pressure</u>	<u>Remarks</u>
June 13. 1967		
8:22 A.M.	473	Starting Pressure
10:08	953	Stop Pressuring
10:23	953	Started Pressuring
11:33	1486	Test on
11:43	1481	
11:53	1478	
12:03	1478	
12:13	1476	
12:23	1475	
12:33	1475	
1:00 P.M.	1475	
1:30	1473	Slight Leak in Dead Weight
2:00	1472 (	0
2:30	1471	
3:00	1472	
3:30	1470	
4:00	1470	
4:00 to 4:20		Working on Dead Weight
4:20	1469	
4:30	1469	
5:00	1469	
5:30	1469	
6;00	1469	
6:05	1469	Start Repressure
6:08	1486	End Repressure
6 <b>:18</b>	1484	
6:28	<b>148</b> 4	
6:38	1484	
6:48	1484	
6:58	1484	
7:08	1484	
June 14, 1967		
8300 A.M.	1478	
9:00	1478	
10:00	1478	
11:00	1478	
11:33	1478	Test Off

Hydrostatic test of 1,185 ft. of 36" pipe, X-100, .250" wall and 10,095 ft. of 36" pipe, X-60, .438" wall. (Line WB-5) Test pressure at dead-weight location 1486. Date of Test - June 13, 1967.

J.

Dead-w <b>bi</b> ght	Pump Stroke	No. of Pump
Pressure	Counter	Strokes
1100	5704	
1110	6056	352
1120	6378	322
1130	6710	332
1140	7041	331
1150	7384	343
1160	7707	323
1170	8031	324
1180	8345	314
1190	8655	310
1200	8983	328
1210	9289	306
1220	9601	312
1230	9907	306
1240	10210	303
1250	10507	297
1260	10820	313
1270	11107	287
1280	11402	295
1290	11691	289
1300	11990	299
1310	12277	287
1320	12570	293
1330	12849	279
1340	13143	294
1350	13478	335
1360	13720	242
1370	13998	278
1380	14258	260
1390	14556	298
1400	14839	283
1410	15116	277
1420	15381	265
1430	15661	280
1440	15949	288
1450	16201	252
1460	16507	306
1470	16771	264
1480	17044	273
1486	17135	91

### PIPELINES AND MAINS TEST DATA

COMPANY DATE Atlantic Seaboard Corporation June 16, 1967 DIVISION DISTRICT STATE Petersburg . . . . W. Va. Elkins LINE NUMBER ACCOUNT OR WORK ORDER NUMBER 36" Line WB+5 ASC Budget 3505 W.O. 359-21 1. A P ÷. PIPE DATA × · LENGTH, FT. PIPE SIZE, NOM. O. D. MILES 36" 1,185 1 0.224 WALL THICKNESS INS. GRADE MINIMUM SPECIFIED YIELD STRENGTH .250 1.1 100,000 PSI 2 X-100 MANUFACTURER PURCHASE ORDER NO. DATE 3 U.S. Steel C-21233 June 25, 1964 TYPE OF LONGITUDINAL SEAM, IF ANY 4 Double Submerged Arc Welded COLD EXPANDED MILL INSPECTED BY 5 Yes 1 DESIGN DATA CONSTRUCTION TYPE "F" ; 6 .72 LONGITUDINAL JOINT FACTOR "E" 1.00 7 TEMPERATURE DERATING FACTOR "T" 1.00 8 DESIGN PRESSURE (See Approved Procedure No. 95, System Standard Policy for Piping Design Pressure) 1000 9 PSIG DATE OF PROOF TEST PROOF TEST DATA June 13 & 14, 1967 LOCATION CLASS TEST MEDIUM 10 1 Water TEST PRESSURE DURATION OF TEST 1529 11 PSIG HOURS 24 COMPANY CONTRACTOR WITNESSED and Hilam BY: TEST ACCEPTED BY INDAN. DATE OF LEAKAGE TEST LEAKAGE TEST DATA (See Form No. G - 10412) TEST MEDIUM TEST PRESSURE 12 PSIG LENGTH OF TEST PRESSURE LOSS 13 PSIG CALCULATED LEAKAGE, CUBIC FEET LOSS PER MILE OF EQUIVALENT 3" AT 100 P S I G 14 CU. FT. CONTRACTOR COMPANY WITNESSED BY: TEST ACCEPTED BY:

NOTE: If the line falls into more than one Construction Type (excepting road, railroad crossings, fabricated assemblies, spans, .) complete separate sheet for each type.

# PIPELINES AND MAINS TEST DATA

COMPANY         Unit					DATE	
STATE V. Va. DIMEUN V. Va. Elkins DIMEUN Elkins ACCOUNT OR WORK ORDER NUMBER ACCOUNT OR WORK ORDER NUMBER S. Steel V. LENGTHORES INS. GRADE VALL TREKESSINS. GRADE VESSION DATA COUNTRUCTION THE "" C LUNGTUDERAL CONT FACTOR "T" 8 1.00 DESIGN DATA DESIGN DATA DESIGN DATA DESIGN FRESURE 9 TLOOD TEST PRESURE 9 TLOOD 10 CONTROL CONT FACTOR "T" 8 1.00 PSIG PROOF TEST DATA 10 CONTROL CONT FREST 10 CONTROL CONTROL CONT FREST 10 CONTROL CONT FREST 10 CONTROL CONT FREST 10 CONTROL CONT FREST 10 CONTROL CONTROL CONT FREST 10 CONTROL CONTROL CONTROL 10 CONTROL CONT FREST 10 CONTROL CONTROL CONTROL 10 CONTROL CONTROL CONTROL 10 CONTROL CONTROL CONTROL 10 CONTROL 10 CONTROL CONTROL 10 CONTROL CONTROL 1	COMPANY Atlantic Seaboard C	orporation		DISTRIC	June 16, 1967	
Live NUMBER         CINING         ACCOUNT OR WORK ORDER NUMBER           SG" LINE WE-5         ASC Bud, 3505 W.O. 359-21           PIPE DATA         ASC Bud, 3505 W.O. 359-21           PIPE DATA         MILES           PIPE SIZE, NON, O.D.         SG"           Value 2000         GRADE           WALL THICKESS INS.         ORADE           2         ASC DUC, SS STEEL           MANUHACTURER         DURCHASS ORDER NO.           3         TYPE OF LONGITUDINAL COM, IF ANY           4         DOUDIO SUBMITING CAM, IF ANY           5         Yees           PIESION DATA           COUNSTRUCTION TYPE 'P''           1.00           TESTEMENTURE CORTING FACTOR 'E''           1.00           PESION DATA           IDENDION THEE 'P''           1.000           PSIO           PROOF TEST DATA           IDENDION CLASS           10         DATE OF PROOF THAT	STATE	DIVISION		DISTRIC	Petersburg	
Bine Model         Asc Bud. 3505 W.O. 359-21           PIPE DATA         MILES           Image: Solar Control of the solar		EIRINS	ACCOUNT OR WORK	ORDER	NUMBER	
PIPE DATA         MILES         MILES           1         56"         51,936         MILES           2         438         GRADE         MILINUM SPECIFIED YIELD STRENGTH           2         438         X-60         Date         60,000 PS1           MAILUTARERS INS.         Steel         593         Jan. 26, 1965         Parts           MANUALUDRER         Steel         593         Jan. 26, 1965         Jan. 26, 1965           MANUALUMER         Submer ged Arc Welded         000 DE XRANDED         Date 26, 1965         Jan. 26, 1965           Vec         Pittsburgh Testing Laboratory         Pittsburgh Testing Laboratory         Pittsburgh Testing Laboratory           DESIGN DATA         CONSTRUCTION TYPE "F"         0         Jan. 26, 1965         Steel           1         LONGTIDUAL JOINT FACTOR "E"         Image: Steel Steel         Steel	36" Line WB-5		ASC Bud, 3505	W.O.	359-21	
HPE SIZE, NOM, O, D.         LENGH, Fr.         MLES         9,84           1         Str.         Str.<	PIPE DATA		and the second			
1     36"     51,936     MINIMUM SPECTRED VIELD STRENGTH 60,000 PS1       WALL THICKNESS-INS AMULACTURER     438     X=50     DATE       MAUHACTURER     438     X=50     DATE       MAUHACTURER     993     Jan. 26, 1965       TYPE OF LONGTUDINAL SEAM, IF ANY Duble Submerged Arc Weided     DATE       COLD EXPANDED     MILL INSPECTED BY       7     Test       DESIGN DATA       COUD EXPRINCE     PT       000STRUCTION TYPE -0"       c     .120       DESIGN DATA       000STRUCTION TYPE -0"       c     .120       LOGGTUDINAL JOINT FACTOR "E"       1.00       PSIG       DESIGN DATA       UNISTRUCTION TYPE -0"       c       1.00       PROOF TEST       1.00       PSIG       DESIGN DATA       June 2,3,4,5,6,7 4 & 8,       1.000       PSIG       DESIGN DATA       June 2,3,4,5,6,7 4 & 8,       DUCATION CLASS       UCATION CLASS       UCATION CLASS       UNTRESED       OONTRACTOR       PSIG   <	PIPE SIZE, NOM. O. D.	LENGTH, FT.		MILES		
will HirkNESS-INS.     UKADE     K-60     60,000 PS1       2     MANUHACTURER     93     Jan. 26, 1965       3     U. S. Steel     93     Jan. 26, 1965       4     Double Submerged Arc Weided     Jan. 26, 1965       5     Yes     Pittsburgh Testing Laboratory       0005 EXEMADED     MILL INSPECTED BY       2     Yes       0005 EXEMADED     Pittsburgh Testing Laboratory       0005 EXEMADED     MILL INSPECTED BY       0005 EXEMADED     Pittsburgh Testing Laboratory       0005 EXEMADED     Pittsburgh Testing Laboratory       0005 EXEMADE     Intervention       1006 EXEMANDE     Intervention       1007     TEMPERATURE DEBATING FACTOR "F"       3     1000       100801000000     PSIG       0000000000000000000     PSIG       000000000000000000000000000000000000	1 36"	51,936		MINIMU	M SPECIFIED YIELD STRENGTH	P. P. Star
2     .438     AUG     PURCHASE ORDER NO.     DATE       3     U. S. Steel     593     Jan. 26, 1965       4     Double Submerged Arc Welded     593     Jan. 26, 1965       COULD EXANDED     MILL INSPECTED BY     Plitisburgh. Testing Laboratory       DESIGN DATA     ODUBION TYPE "P"       6     .72       1.00     TEMPERATURE DEATING FACTOR "P"       8     1.00       7     1.00       9     1.00       9     1.00       9     1.00       9     1.00       9     1.00       9     1.00       9     1.00       9     1.00       9     1.00       9     1.00       9     1.00       9     1.00       9     1.00       9     1.00       10     DESIGN PRESSURE       9     1.000       11     TEST PATA       12     100       13     10       14     Matter       14     12       12     100       13     10       14     10       14     10       14     10       14     10 </td <td>WALL THICKNESS - INS.</td> <td>GRADE X-60</td> <td></td> <td></td> <td>60.000 PSI</td> <td></td>	WALL THICKNESS - INS.	GRADE X-60			60.000 PSI	
a MUCROUNAL S. Steel     593     Jan. 26, 1965       TYPE OF LONGITUDINAL SEAM, IF ANY     0       4     Double Submerged Arc Weided       COLD EXPANDED     MILL INSPECTED BY       5     Yes       0     Pittsburgh Testing Laboratory       0     DESIGN DATA       0		A-00	PURCHASE ORDER NO.		DATE	
TYPE OF LONGITUDINAL SEAM, IF ANY       4     Double Submerged Arc Weided       COLD EXEMNED     MILL INSPECTED BY       5     Yes       Pittsburgh Testing Laboratory       DESIGN DATA       CONSTRUCTION TYPE "F"       6     .72       CONSTRUCTION TYPE "F"       7     1.00       TEMPERATURE DENTING FACTOR "E"       7     1.00       TEST MATA       DESIGN PRESSURE       9     1,000       PROOF TEST DATA       10     TEST PRESSURE       10     1       UDATE OF PROOF TFSI       1967       June 2,3,4,5,5,6,7 & 6,       ODATE OF PROOF TFSI       DESIGN PRESSURE       10     1       TEST MATA       ULCONTION CLASS       TEST PRESSURE       ODATE OF PROOF TFSI       1967       DURATION OF TEST       ULCONTINCTOR       WITHESED       OUNTRACTOR       OUNTRACTOR       DURATION OF TEST       ULCONTRACTOR       OUNTRACTOR	3 U. S. Steel		593		Jan. 26, 1965	
4     Double Submerged Arc Weided       COLD EXPANDED     MILL INSPECTED BY       PITTSburgh Testing Laboratory       DESIGN DATA       CONSTRUCTION TYPE "F"       d       CONSTRUCTION TYPE "F"       d       1.00       TEMPERATURE DERATING FACTOR "E"       9       1.00       PROOF TEST DATA       10       DESIGN PRESSURE       9       1.00       PROOF TEST DATA       10       11       1606       PSIG       12       13       14       15       14       15       16       16       17       18       19       10       10       10       11       11       12       13       14       14       15       15       16       16       17       18       19       10       10       10       11       12       13       14       14        15	TYPE OF LONGITUDINAL SEAM, IF ANY					
COLD EXPANDED     MILL INSPECTED BY       S     Yes       DESIGN DATA       CONSTRUCTION TYPE "F"       G     .72       LONGITUDINAL JOINT FACTOR "E"       7     1.00       TEMPERATINGE FACTOR "T"       8     1.00       DESIGN PRESSURE       9     1,00       PROOF TEST DATA       LOCATION CLASS       10       TEST PRESSURE       11       1606       PSIG       CONTRACTOR       WITNESSED       DOTRACTOR       PROOF TEST DATA       10       11       1606       PSIG       CONTRACTOR       WITNESSED       DONTRACTOR       PSIG       CONTRACTOR       PSIG       CONTRACTOR       PSIG       CONTRACTOR       PSIG       CONTRACTOR       DATE OF LEAKAGE TEST       TEST ACCEPTED BY       Accepted BY       LEAKAGE TEST DATA       IEENT MEDIUM       TEST ACCEPTED BY       LEAKAGE TEST DATA (See Form No. G - 10412)       TEST PRESSURE       PSIG       CONTRACTOR       PSIG       CONTRACTOR       PSI	4 Double Submerged Arc	: Welded				1000 - 1000 
5     Yes     Principal restring Labor and Y       DESIGN DATA     CONSTRUCTION TYPE "F"       6     .72       1.00     1.00       TEMPERATURE DERATING FACTOR "E"       2     1.00       DESIGN PRESSURE       9     1.00       PROOF TEST DATA       Location class       10       1       10       1       10       1       10       1       10       1       10       1       10       1       10       1       10       1       10       1       10       1       10       1       10       1       10       1       10       1       10       1       10       1       10066       11       10067       12       13       14       14       14       14       14       14       15       16        12	COLD EXPANDED		MILL INSPECTED BY		. Laboratory	
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13     TSTG       14     CALCULATED LEAKAGE, CUBIC FEET LOSS PER MILE OF EQUIVALENT 3" AT 100 P S I G     CU. FT       WITNESSED BY:     CONTRACTOR     COMPANY       TEST ACCEPTED BY:     CONTRACTOR     COMPANY	LENGTH OF TEST		PRESSURE LUSS			PSIG
CALCULATED LEAKAGE, CUBIC FEET LOSS PER     CU. FT.       14     MILE OF EQUIVALENT 3" AT 100 P S I G     CU. FT.       WITNESSED     CONTRACTOR     COMPANY       BY:     CONTRACTOR     COMPANY	13					1010
WITNESSED BY:     CONTRACTOR       TEST ACCEPTED BY:	CALCULATED LEAKAGE, CUBIC FEET LOSS F	'ER				CU. FT
WITNESSED       BY:       TEST ACCEPTED BY:	CONTRACTOR		COMPANY			
TEST ACCEPTED BY:	WITNESSED BY:					1.1
TEST ACCEPTED BY:						
	TEST ACCEPTED BY:					

NOTE: If the line falls into more than one Construction Type (excepting road, railroad crossings, fabricated assemblies, spans, ...) complete separate sheet for each type.



CAS TEST OF 53,167' OF LINE WB-5-36" 1965 CONSTRUCTION A.S.C. BUD. 3505, W.O. 359-21 TEST TIME: 2:00 pm - 2:00 pm ( 24 HR.) DATE OF TEST: NOV. 29-30, 1965 AVE, DEAD WT. TEST PRESSURE = 884 pS19. DESIGNI PRESSURE: 1000 PS14. (800 PS14. FUR WINTER, 1965-66)

	PTCARAGE FACTO	OFFICE
	SIGNED S.T. Collin	CHART IS REMOVED
	REMARKS	ONT WHEN FILLED
W.d 00:2 14 5 961	- 02 - 11 - 30 -	TO BE
	SIGNED Z'L' Coffere	LACED
W.d 00:2 14 59 61	26-11 ON TRAHD	CHART IS
24-4	AMAN NOITATS	UNT WHEN
EATE .	CTELES MOORE FIELD	TO BE

FBTT .ON DNITNING XDAB DROBX03

□ Immediate action desired	Date 12-17-64
ТО	
Mr. Forrest	Robinson
FROM	
Jack G. Brow	n
With reference to the at	ached:
Take charge	
Approval required	Return to me
Signature required	Return to me
Prepare reply for m	y signature
Reply. Send me cop	у
Advise status	
Review and contact	me this A.M P.N
Comments desired	
Recommendations of	esired
Note and return	
Note and file	
For your information	l.
Remarks:	
This has bee	n approved for payment.
cm1	

	AME ADV	RICAN ANCED	MACHINE & I PRODUCTS GR	FOUNDRY C OUP, Greenw	<b>омі</b> ich,	PANY Conn.	*		
	REMIT	το: <b>261</b>	Madison Aven	ue, New York	16,	N.Y.			
CUSTOMER'S OR P.O. 352	DER NO. &	2 DATE	REQUISITION OR CONTRACT NO.	CUSTOMER CODE	J	REFER TO OB ORDER NO	).		
INVOICE N	<b>0.</b> 143	7	TERMS Net 30	Days	INVC Dec	DICE DATE <b>27, 1964</b>			
то	Atla 1700 Cha	ntic Sea Mac Co rleston	aboard Corpora orkle Ave., S. 25, West Virgi	tion E. nia		-			
SHIPPED TO A	ND DESTIN	ATION					BALANCE OF WILL FOLLO THIS SHIPME COMPLETES	ORDER W. NT YOUR OR	DER
•			r					J 1.0.D.	
JOB ORDER NO.	QUAN ORDERED	TITY SHIPPED	PART NUMBE (INDEX) RAT	R - DESCRIPTION TED (CLASS D	) )	UNIT LIST PRICI	AMOUNT		TOTAL
			Use of AMF Columbia Gas line for perio 1964 thru 10	Welder on s System Pipe od of 23 Octob December 196	er 64.				
			(Reference yo	our P.O.#352	52)				\$12,970.70
			Charge: In Research Acct. No.	nstalling Pipe-Line W 186-1-879.	B-5		-		
						2. •		p.	
		1							
	8 > *		6		-		* 1	a.	
"We hereby cer	tify that the	ese goods w	vere produced in comp	liance with all applic	able 1	requirements of	of sections 6, 7.	and 12 of	the Fair Labor

articles ordered were produced in compliance with any and all applicable provisions of the Walsh-Healy Public Contracts Act, the Eight Hour Law and the Davis-Bacon Act." "It is the Seller's policy to conform with all applicable ceiling price regulations." TERMS: NET CASH 30 DAYS, UNLESS OTHERWISE SPECIFIED ABOVE - NO CASH DISCOUNT - PAYABLE IN U.S.A. DOLLARS

ADDITIONAL COPIES

COPIES (CUST.)

ORIGINAL

TERMS: NET CASH 30 DAYS, UNLESS OTHERWISE SPECIFIED ABOVE - NO CASH DISCOUNT - PAYABLE IN U.S.A. DOLLARS ALL GOODS F.O. B. OUR FACTORIES. MAKE NO DEDUCTIONS FROM THIS INVOICE: IF INCORRECT RETURN AT ONCE.

1 PINK

1 POSTING

1 COLLECTION

1 SHIPPING

	AMERIC ADVANC	CED : 261	MACHINE & FO PRODUCTS GRO Madison Avenu	DUNDRY C UP, Greenw e, New Yorl	<b>OM</b> ich, <b>(16,</b>	PANY Conn. N.Y.			
CUSTOMER'S ORDE P. O. 35252	R NO. & DA	TE	REQUISITION OR CONTRACT NO.	CUSTOMER CODE	J	REFER T OB ORDEI	'O R NO,		
INVOICE NO.	1437		TERMS Net 30	Days	INVO De	DICE DATI c 7, 19	е 64		
то	Atlanti 1700 Ma Charle	c Sea ac Co ston	aboard Corporati orkle Ave., S. E 25, West Virgini	on • ia			]		
SHIPPED TO AND	DESTINATIO	ON		- 8				ALANCE OF ORI ILL FOLLOW. HIS SHIPMENT OMPLETES YOU TE SHIPPED F.	DER R ORDER O. B.
JOB ORDER	QUANTITY	Y	PART NUMBER	- DESCRIPTION		UNI			
NO. OF	DERED SHI	IPPED	(INDEX) RATE	D (CLASS I	oiv.)	LIST P	RICE	AMOUNT	TOTAL
ž.			Use of AMF W Columbia Gas line for period 1964 thru 10 D	elder on System Pipe of 23 Octob ecember 19	er 64.				
			(Reference you	r P. O. #352	52)			1	\$12,970.70
		5 e			14		ă.		
		.)	Charge: Ins Research P Acct. No.	talling ipe-Line W 186-1-879.	B-5	(*1	8		
								8	
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			G						

Standards Act, as amended, and of regulations and orders of the Administration issued under section 14 thereof. We also warrant that the articles ordered were produced in compliance with any and all applicable provisions of the Walsh-Healy Public Contracts Act, the Eight Hour Law and the Davis-Bacon Act." "It is the Seller's policy to conform with all applicable ceiling price regulations."

TERMS: NET CASH 30 DAYS, UNLESS OTHERWISE SPECIFIED ABOVE - NO CASH DISCOUNT - PAYABLE IN U.S.A. DOLLARS ALL GOODS F.O.B. OUR FACTORIES. MAKE NO DEDUCTIONS FROM THIS INVOICE: IF INCORRECT RETURN AT ONCE.

ADDITIONAL COPIES

COPIES (CUST.)

ORIGINAL

1 SHIPPING

1 PINK . 1 POSTING 1 COLLECTION

FORM CS 1-257 -P CED (8-61)	COLUMBIA GAS WORKING FUND	SYSTEM VOUCH	IER					DA	TE Je	an.	11,65	
TO Monarch Mi	lls, Inc.											
ADDRESS P. O. Box	126 - Petersburg, West Vin	ginia	-							\$_	147.5	9
ISSUED FOR Supplie	8										147	59
								-			1	
Record	h Line Pine WB-5 - Account	: 186-0	- 2- {	31							1.5	
TO BE USED FOR REDEGIN	a mane s ape an 5 mooden	. 200 0										
PREPARED BY	DECEMED OF	1					<u>d</u>		ineli ineli			*
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anot Doule	Atlantic Seaboard Corp.co.		ST. E	LD	GEN. LED.	AUX	INT.	SUB.	OTHER	CODE	AMO	UNT
APPROVED BY	and 59/100					1						10. sa
APPROVED BY	SIGNED										in it was	
STATEMENT 6 SUB 9 NUMBER NO.	Paid by Ck. No. 10126											

Monarch Mills, Inc.

BUILDING MATERIALS - FARENAS' SUPPLINS RESODELING NEW CONSTRUCTION TELEPROPER CERCLE 5-4611 - 5-8681 P.O. BOS 186 FRITERBURG, W. VA. SUPER

ATLANTIC SEABOARD CORP. FETERSTURG, M. VA.

#### (ba)

TERMS 30 DAYS

INTEREST CHARGED ON PART DUE ACCOUNTS

				A DESCRIPTION OF A DESC
067 31 64	60 FT 1/2" ROPE	3.00		2.46
NOV 3 SA	I PAINT BRUSH PAINT BRUSH TAX A RLS 939-12-12 WIRE AO T' STEEL POSTS RL. GARBED WIRE SF STAPLES 6 STEEL BRUSHES TAX LINE WB-S ACCT NO. 196-0-2-01 W.P.DIGML 100 FT LATTICE RX LINE WB-S ACCT 106-0-2 W. P. DIEHL JR	1.99 .79 .00 50.00 9.00 9.00 .30 3.34 4.03 .30 3.34 4.03 .10		130.07
NOV 12 6A I	1 BH. 1/2X4X8 AD PLYBOO LABOR BX BY 1 RL 939-12-122WIRE BY TAX IO PIPE FITTINGS TAX	3,89 .75 .20 17.65 0 .53 0 .97		13948
NOV I T GA	BY CHECK 1 1/8x8x8 PLYBOOD MILLING RX	3.20 30 .11	2.45	137.22

PLEASE KEEP THIS INVOKE - WE FORTIVELY DO NOT ITEMIES AGAIN

4

Monarch Mills, Inc.

BUILDING WATANAAA PATANKA ASAFI PAR MANGGALANG NAR CONSTRUCTION TELAVOLARE CONSTRUCTION A DIGATES PRICEMBER W. VA. 1999

## ATLANTIC SEABOARD CORP. PETERSBURG, W. VA.

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107 2 7 64	1 # 200 NAILS	.14		
	IF ISO NAILS	.12 .01		141.94

PLEASE KEEP THIS INVORCE -- WE POSITIVELY DO NOT TURKIZE AGAIN

Monarch Mills, Inc.

EDILORO HATIMALO - VARIASSE AVERADO ARMORILINO - HEN CONSTRUCTION TREPPONDE CREATE C-4811 - 0-0022 P.O. 802 186 - PETTRUBURO, W. VA. 20145

ATLAUTIC SEARCARD CORP. PETERS AUG, V. VA.

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\$16 G	i 50FT steel tape Tax	5,49 .16		142.94
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	Γ	MONARCH	MILLS	, IP	NC.		
	R. H. ALT	BOX 126 PETERS CIRCLE 3-4511	BURG, WEST VIR	GINIA 2 CLE 3-5	6747 5 <b>61</b>	ROSWELL H. AL	.т.
	BUILDING MATE	RIALS FARM & GARD WHOLESALE	EN SUPPLIES and RETAIL	B	UILDING CON	TRACTING	
ESMAN	CUSTOMER ORDER NO.	WHEN TO SHIP	PICK UP			DATE 11/4/	64
NO	TE-THIS IS A JOINT ACCOUNT	antic Se	hory	0	т	ERMS: 30 DAY	SNET
s O L	r. & Mrs.				s	JOB	
					Р Т		
SH	CONTRACT CHARGE	ON ACCT. MDSE. RETI	D. INTEREST C	CHARGE	D ON PAST DUE	ACCOUNTS	
QUANTITY	DES	CRIPTION	QUANT SHIPI	PED	PRICE	AMOL	JN T
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		R. H. ALI		CIRCLE 3-4	511	CIRCLE	3-5561	ROSWELL H. ALT.
		BUILDI	NG MATE	RIALS I	FARM & GARDE	N SUPPLIES	BUILDING CON	ITRACTING
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		R.H.ALT		BOX 126 CIRCLE 3-4	PETERS 4511	BURG,	WEST VIRGIN	NIA 26747 E 3 <b>-5561</b>		ROSWELL H. A	LT.
		BUILD	ING MATE	RIALS	FARM & GARD	EN SL		BUILDIN	IG CON	TRACTING	
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	R.H.ALT	BOX 126 PETERSBURG	WEST VIRGINIA 2	6747 5 <b>561</b>	ROSWELL H. ALT.
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	R. H. AL	т	BOX 126 CIRCLE 3-4	ARCH I Petersi	<b>MILLS, I</b> BURG, WEST VIRGINIA CIRCLE 3-	<b>NC.</b> 26747 5561	ROSWELL H. ALT.
	BUILI	DING MATE	RIALS	FARM & GARDE WHOLESALE	EN SUPPLIES E	BUILDING CONT	RACTING
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	CIRCLE 3-4511	CIRCLE 3	-5561	SWELL H. ALT.
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N.H.AT     BOX 123     PETERSBURG, VEST VIRGINIA 25747     DOWELL H., ALT.       CIRCLE 3.4511     CIRCLE 3.5561     DOWELL H., ALT.       BUILDING MATERIALS     RAM & GARDEN SUPPLIES     BUILDING CONTRACTING       WOLESALE and RETAIL     BICK UF     DOWEL H., ALT.       BUILDING MATERIALS     RAM & GARDEN SUPPLIES     BUILDING CONTRACTING       NOTE-THIS IS A JOINT ACCOUNT     DECK UF     DOWEL H., ALT.       Mr. & Mrs.     Allowed ut     Deck UF       Mr. & Mrs.     Description     Output       Mrs. & Mrs.     Description     Output       Description     Output     Price     Andown       Mrs. & Mrs.     Description     Output       Mrs. & Mrs.     Mrs.     Mrs.       Mrs. & Mrs		MONARCH M	ILLS, II	NC.		
CIRCLE 3-4511 CIRCLE 3-5561 BUILDING MATERIALS FARM & GARDEN SUPPLIES BUILDING CONTRACTING WHOLESALE and RETAIL  ALLEMAN EUSTONES DESEND  NOTE-THIS IS A JOINT ACCOUNT  NO. 8. Mr.  ALLEMAN CONTRACT CHARGE ON ACCT.  DESCRIPTION		R. H. ALT BOX 126 PETERSBUR	ROSWELL H. ALT.			
BUILDING MATERIALS     FARM & GARDEN SUPPLIES     BUILDING CONTRACTING       WHOLESALE and RETAIL     UATE     Intervention     Intervention       NOTE-THIS IS A JOINT ACCOUNT     Intervention     Intervention     Intervention       Mr. & Mrs.     Intervention     Intervention     Intervention       Building Contract     Intervention		CIRCLE 3-4511	5561			
ALESMAN CUSTOMES ORDER NO. WHEN TO BHIP PIECUP DATE A-23 THE A-23		BUILDING MATERIALS FARM & GARDEN WHOLESALE and	SUPPLIES B	UILDING CON	ITRACTING	
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	R.H.ALT	CIRCLE 3	PETERSBUI	RG, WEST VIRGINIA	6747	ROSWELL H. AL	.т.
	BUILDING MAT	ERIALS	FARM & GARDEN	SUPPLIES B	UILDING CON	TRACTING	
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	R.H.ALT	BOX 126	PETERSBURG	, WEST VIRGINIA 2	26747	ROSWELL H. AL	т.	
		CIRCLE 3-4511		CIRCLE 3-5	5561			
	BUILDING MAT	ERIALS FARM	LESALE and	RETAIL	UILDING CON	TRACTING		
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Property - W. O. W.0 359-21 Bud 3505 - Line WB-5

X-Ref Engineering + Planning Color. - Catte Prot (Inspection + Festing) February 15, 1966

Mr. Leo J. Payne Mr. J. Frank Dickerson

CROIN

Gas Test Line WB-5

Attached are the Pipelines and Mains Test Data Sheet with recording charts for the gas test of 36" Line WB-5 from Moorefield to Route 220.

This section of line was gas tested due to impending delivery requirements and the possibility of sub-freezing weather interfering with a standard hydrostatic test. The contractor spotted a portable gas compressor at Moorefield to boost test gas from the 26" System to a final test pressure of 884 Psi.

A hydrostatic test to yield will be put on this section during 1966 by the contractor, Carl E. Smith.

Until further test, this section of 36" Line is available for operation at 804 Psi.

J.Frank Dickerson

cc: Mr. R. E. Lynn WB-5 Test File
3

1

## PIPELINES AND MAINS TEST DATA

LOW	ATLANTIC SEABOARD CO	PORATION		DATE	bruary 16, 1966		
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INC	NUMPED	Elkins	Antone Int	and there	C.F.		
	WB-5		Budget 3505 W. O. 359-21				
PIP	E DATA						
1 F	PIPE SIZE, NOM. O. D. 36"	8 *	MILES 9.7	9.72			
2	WALL THICKNESS - INS. .438	GRADE X-60		MINIMUM SPECIFIE	O YIELD STRENGTH		
3	WANUFACTURER U. S. Steel		PURCHASE ORDER NO		an. 26, 1965		
1	TYPE OF LONGITUDINAL SEAM, IF ANY Double Submerge	d Arc	e to provide press		And the State		
5	COLD EXPANDED Yes		MILL INSPECTED BY P. O. 4381	Pittsburgh Testing Co. March 19, 1965			
DES	SIGN DATA	Service Alexandra Service	San Parat State	and the second second			
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T	EMPERATURE DERATING FACTOR "T"	The Barrier of		an anna an			
D	DESIGN PRESSURE	(See Approved Procedure	No. 95, System Standar	d Policy for Piping Des	ign Pressure)		
PRC	OOF TEST DATA	The West		DATE OF I	mber 30, 1965		
L	1	and the first state	TEST MEDIUM Gas				
T	EST PRESSURE		DURATION OF TEST	74	11		
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	BY: Carl L. Smit		FTOAd	UITICN			
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LA	KAGE IEST DATA (See Form I	No. G - 10412)	TECT DECOUDE				
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C/ M	ALCULATED LEAKAGE, CUBIC FEET LOSS	PER	San San Jan		FST		
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NOTE: If the line falls into more than one Construction Type (excepting road, railroad crossings, fabricated assemblies, spans, .) complete separate sheet for each type.

- \* 1185 of 36", X-100 Experimental pipe laid in 1964 was also tested.
- \*\* Line designed for 1000 Psi, this test qualifies it for temporary operation at 804 Psi until retest during 1966.









FOXBORD BACK PRINTING NO. 1781

AN BONE

TO BE FILLED	STATION NUMBER MOORE FIELD GATE				
CHART IS PLACED	CHART ON 11-29 SIGNED 5. T. Collies	19.65 AT 2:00 P.M.			
TO BE FILLED OUT WHEN CHART IS	CHART OFF 11 - 30 - REMARKS	19 6 5 AT 2:00 P.M.			
OFFICE	AVERAGE FAC	TOR			

DESIGN DEESSARE: 1000 DEIS (800 DEIS 1- 1-08 MINLER' 1392-98) UNLE DE LEST: NON 55-30' 1392 DULLE OF LEST: NON 53-30' 1392 DULLE OF LEST: NON 53-30' 1392 DULLE OF LEST: NON 53-30' 1392 DULLE OF LEST: NON 56-30' 1392 DULLE OF LEST DULL

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1965 CONSTRUCTION

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"92-5-8M ENT 20, L91ES 20 1521 587

## Appendix B. Pipe Section Details







Appendix C. Mechanical Test Specimen Cut Plans

th welds must be aligned axially with the weld center line. thout welds are aligned parallel with (reference lines provided on pipe) or perpendicular dis.	n is unimportant. Sprues must be f the specimens. centerlines marked on plate.		9.25 9.25 9.25 9.25 9.25 9.25 9.25 9.25		DADT A/TEW.	PISI	Scale: In View SHEET:1 of 1 CREATION DATE: 8/11/2021 REVISION: Contact: Dach Woake - timdach@nict dov 303-968-8823	The information contained on this drawing shall be considered intellectual property owned by NIST. Authorized receipt and review of this information implies consent to non-disclosure of trade secrets. Disclosure to third parties is unauthorized without prior written permission from NIST.
Specimens w and centrally Specimens w the pipe axis to the pipe ax	<ul> <li>Kerf dimensic</li> </ul>		A9 A10 A11 3.00	Flat Side	DP/JECT.		QTY REQUIRED: 1	DEFAULT UNITS: Inches       DEFAULT TOLERANCE: ±0.005"-±1       DEFAULT FINISH: < 63 microinch
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Appendix D. Metallurgy Specimen Locations




































