Final Report

U.S. DOT
Pipeline and Hazardous Materials Safety Administration

Competitive Academic Agreement Program

Advanced Nondestructive Characterization of Pipeline Materials

L. J. Bond, B. Engle and L. Smart

Iowa State University

DTPH56-13-H-CAAP07

12/31/2015

SUMMARY

This project set out to seek to provide a proof-of-concept demonstration for the characterization of mechanical properties in pipeline steels, such as strength and toughness, using nondestructive measurements. The goal being to seek results that could serve as the basis for further integrating nondestructive characterization technologies into inline inspection tools.

Working with industry partners, an initial sample set was obtained for study. Preliminary nondestructive measurements were performed, but results were inconclusive; there were found to be many variables changing between samples which proved to make it impossible to establish correlations between pipe-sample data and results inferred from the nondestructive results. By analyzing a database of sample properties collected by an industry partner it was shown that common 'rules-of-thumb' assumptions relating pipe parameters, such as those relating to yield strength, chemical content and even gran size were poorly reflected or not seen in the data for pipe samples that had been reviewed, any of which were "good material" removed from near failure or other intervention sites. From the literature it is found that property relationships are generally reported where samples were obtained from one mill, with one chemistry, and with basically similar process parameters, which were varied in some controlled fashion, such as to vary a single parameter such as grain size. These observations imply one of two conclusions: either the samples considered in the data base were not representative of the general set of pipeline steels, or that these rules-of-thumb relationships repeated in the literature are in fact poorly reflected for the general set of pipeline steels found in service.

In seeking to unravel the complex property and process parameter issues a small set of samples were obtained from the Colorado School of Mines, which were X52 and were post-processed to change just grain size and pearlite content. NDT tools were then applied to these samples. It was shown that when using measurements on a small, controlled sample set with different heat treatments applied to the same base material causes variation in the nondestructive measurement results. This highlights the need to understand which microstructural variables affect the nondestructive results. Some correlations were observed, but it is clear that no one measurement will provide the desired mechanical property relationships to NDT measured metrics. Some trends were observed that indicate that a first step needs to be to simplify the problem by considering NDT applied to families of samples with fixed chemistry, for example. It is recommended that further work on this topic be considered under two topics: 1.) the relationship between the chemical composition and microstructure with the strength and toughness properties of interest, and 2.) the effects of key microstructural parameters on nondestructive measurements. The first study should seek to determine that if the chemical composition and microstructure of a steel is fully known within a random set of pipeline steels covering the range of those currently in service, can the strength properties be estimated? The second study should then use a series of controlled investigations, with well characterized samples with known processing parameters, to investigate the effects on NDT measurements of individual microstructure parameters, and only then expanding data collection towards a multivariate problem where several parameters in the steel samples are allowed to vary.

TABLE OF CONTENTS

1. INTRODUCTION 4

2. BACKGROUND 4

1. Steels 4
2. NDE for Material Characterization 5

3. APPROACH 9

4. TASK 1: OBTAIN SAMPLES 10

1. Initial Pipe Samples 9
2. Kiefner Sample Database 12

5. TASK 2: RELATIONSHIP BETWEEN MATERIAL AND MECHANICAL PROPERTIES 15

1. Linear Modeling: Mechanical/Material Property Relationships 20

6. TASK 3: NONDESTRUCTIVE MEASUREMENTS 25

1. Pipe Samples 25
2. Controlled Samples 27

7. TASK 4: CONCLUSIONS AND RECOMMENDATIONS 32

1. Material/Mechanical Property Relationships 32
2. NDE/Material Property Relationships 32

8. CONCLUSION 33

PROJECT PUBLICATIONS AND PRESENTATIONS 34

REFERENCES 34

1. INTRODUCTION

The United States has a vast network of natural gas pipelines totaling over 300,000 miles. A large portion of this pipeline infrastructure was installed in the 1950s/60s/70s. The local values for strength and toughness parameters of this aging pipe infrastructure needs to be known so as to be able to asses if it is possible to expect to continue to be operate these lines safely at some specified operating pressure. This need is highlighted by the effects of events such as the San Bruno incident [NTSB 2011]. This incident highlighted the need to know pipe fabrication process (i.e. is there a seam weld), pipe condition and characteristics of defects, as well local strength properties.

Various in-ditch tests, such as hardness or indentation tests, can be performed on pipelines by excavation to get access to the external surface of the pipe. Currently the only way to reliably assess the strength and toughness properties of pipeline steels is to remove a section from an in-service pipeline and test it destructively. This method has downsides, not least the need for pipe access and it can involve a hot-tap to remove a sample. Such activities do have the risk of introducing flaws as a result of the cutting process.

In-line inspections tools are commonly used to assess pipe condition. These tools are put into the pipeline and propelled by the contents of the pipe while collecting data with a variety of electromagnetic and ultrasonic sensors. These in-line assessment tools have the advantage of not needing excavation to get access, and can provide inspections of 10's to 100 km in a single run. Large quantities of data are recorded that are analyzed to identify metal loss and flaw/crack detection.

The main objective of this project was to determine if it is possible to NDE tools to estimate the local values for pipe mechanical properties, specifically:

- yield strength – (strength until plastic deformation starts)
- tensile strength – (strength until failure)
- toughness – (energy absorbed before failure)
- ductile-to-brittle transition temperature – (temperature at which a material loses ductility and becomes brittle).

It also had the long-term goal of identifying a sensor or multi-sensor modality that can be integrated into an in-line inspection tool that utilized these measurements to estimate the strength and toughness of the material. Such an advance would provide data to complement what is currently obtain and used for detecting metal loss and other flaws.

2. BACKGROUND

2.1 Steels

Steels are used in a variety of industries for various purposes [Krauss, 2005, Samuels, 1999]. Steelmaking is a complex process that has evolved over time [Gray and Siciliano, 2009]. Various alloying elements and processes have been used historically to produce steels that meet certain standard grades, such as API X52. These standards specify a *minimum* yield strength (not the

actual strength) and place bounds on certain alloying elements, such as carbon. The grade of a pipeline steel helps define the expected performance of the steel, as well as giving some indication of the alloying elements used, but it does not completely describe the steel.

A summary of steel manufacturers shows that pipe from standard steel mills has been made since the early 1900s, resulting in a variety of grades of pipes, and a variety of methods of welding and joining the steel plates have been employed [Kiefner and Clark, 1996]. Almost 50% of US pipelines currently in use were installed between 1950 and 1970 when the manufacturing processes were less refined and for which, when assessments are needed, records may now be incomplete [Kiefner and Rosenfeld, 2012].

Different manufacturing processes can result in variation in properties within a specified grade of steel. For instance, the relationships between the thermo-mechanical processing effects to the yield strengths have been developed in modern steel-making [Zhao et al. 2002]. The relationships indicate that the start and finish rolling temperatures, as well as the rate of cooling and the finish temperature are strongly correlated to the resultant yield strength ($R = 0.94$), which is a parameter that is a direct result of the microstructure.

2.2 NDE for Material Characterization

The most accurate determination of yield and tensile strength is through destructive testing, although the procedures themselves have inherent variability and only provide data for the sample actually tested. For a pipe sample that is initially curved depending on the type of procedure performed and the flattening method used for a tensile strap test, a standard deviation of the yield strength value of up to 26.2 MPa (3800 psi) has been seen in a recent study [Cooreman et al. 2014]. This inherent variability found in destructive testing needs to be considered when determining what NDE methods could potentially be used for material characterization in lieu of destructive methods.

Nondestructive evaluation (NDE) is used in many applications for flaw detection and material characterization [Raj et al. 2003]. Ultrasonic, electromagnetic, and radiographic measurements have uses for detecting flaws in the aerospace and energy industries. In-line inspection tools for flaw and metal loss detection often utilize NDE technologies. Magnetic flux leakage has recently been used with success to determine the location of hard spots, a material deviation from the original properties which are generally a result of manufacturing processes [Belanger and Barker, 2014]. However, the hardness value of these identified locations is not yet quantified by current technology. Eddy current technology has been used recently to estimate the grade of the pipe by using a pre-magnetization technique to increase penetration depth needed for the thickness of pipeline steel, with typical values ranging from 0.188 inches to 0.675 inches or greater for heavy wall pipe. Preliminary results from pull tests show that accuracies of ±40 MPa (5801 psi) may be achievable, but more testing appears to be needed to verify these results [Molenda and Thale, 2014]. In-ditch techniques currently in use include hardness measurements, chemical content assessments through x-ray fluorescence (XRF) or optical emission spectroscopy (OES), and microscopy for determining microstructural characteristics. This current project sought to use and build on existing NDE knowledge to characterize the pipeline steels and use the resulting data to establish correlations to estimate strength and toughness.

Ultrasonic measurements use propagating elastic waves to characterize materials and detect flaws [Thompson 1983, 1996]. The waves scatter when they encounter a mismatch in acoustic impedance, which could be a flaw (void, inclusion, or crack) or the boundaries between individual grains in the microstructure. The microstructural scattering has potential for characterizing microstructure in pipeline steels. The energy lost from a propagating ultrasonic wave, called the attenuation, can be measured and is largely dependent on the scattering from the individual grains. The energy scattered from the microstructure can also be directly measured, in what is referred to as a backscatter or grain noise measurement. The velocity at which the ultrasonic wave propagates can also be measured, and this is related to the elastic constants of the material [Ensminger and Bond, 2011].

The collected data for measuring ultrasonic velocity and attenuation often resembles that shown in Figure 1. These data are voltage (amplitude) – time traces. In a flat sample with a pulse-echo measurement the transducer sends a signal normal to the surface and an ultrasonic wave enters the sample and it is reflected by the back surface. The wave then gives multiple reflections as it reverberating within the sample. At each front surface interaction, some of the energy exits the sample and returns to the transducer, allowing for detection of each back wall reverberation. The transit time between echo's is controlled by the sample thickness and wave velocity.

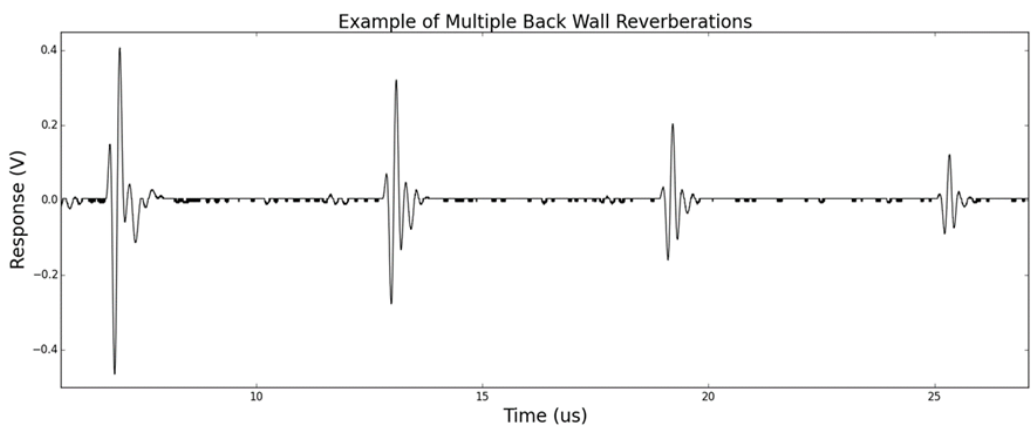


Figure 1. Multiple back wall reverberations of an ultrasonic wave propagating through a sample.

The arrival times of the reverberations as shown in Figure 1 can be used in conjunction with the measured wall thickness to provide an estimate of the ultrasonic velocity in the sample. Figure 2 shows this process, where the slope of the line is used for the estimate of velocity. Examples of error due to wall thickness measurement uncertainty are shown, as upper and lower bounds).

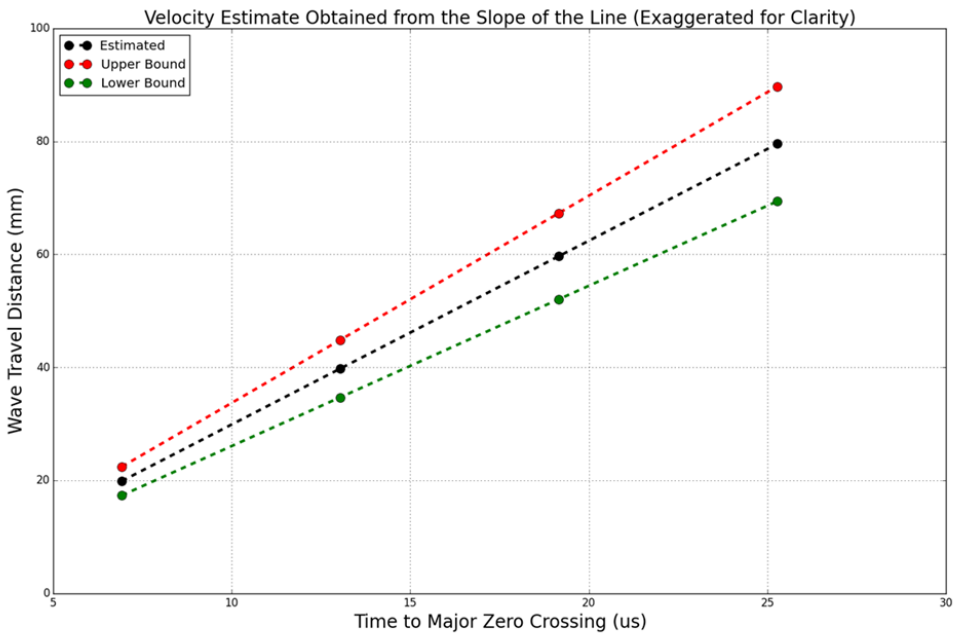


Figure 2. Wave travel distance inside a sample plotted against the measured time of signal arrival. The slope is an estimate of the ultrasonic velocity of the material.

To estimate the ultrasonic attenuation the data are considered in the frequency domain (each ultrasonic signal contains a band of frequencies). The individual signals are windowed and a Fourier Transform applied to give the spectrum. A scaled logarithm of the ratio of these spectra is then related to the attenuation of the sample material. An example of the ratios of the spectra of these back wall reverberations which are used is shown in Figure 3.

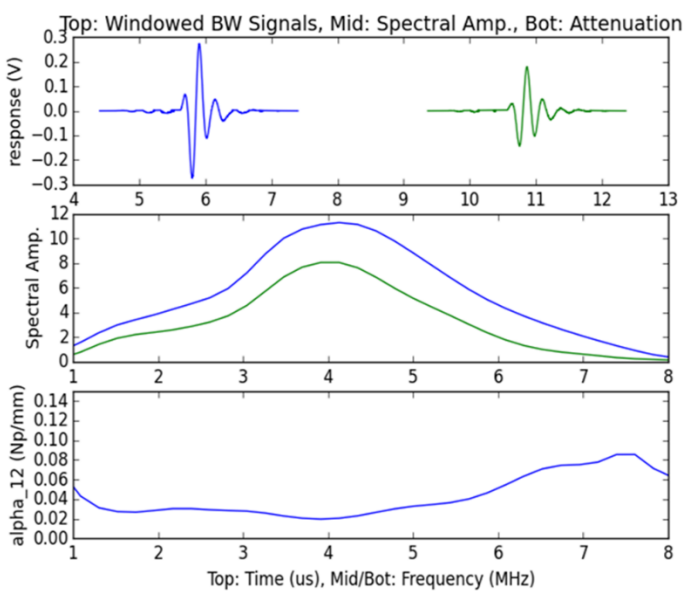


Figure 3. Example of attenuation curve determination. Back wall signals (top), spectrum of each (middle), scaled logarithm of ratio of spectra (bottom).

Ultrasonic attenuation and grain size determination has been discussed in a number of publications and software to perform the data analysis has been demonstrated and validated in a series of studies for the aero-space engine community [Margetan et al. 2014]. Figure 4 outlines the process for measuring ultrasonic backscatter. The transducer in water in pulse-echo gives the sort of response shown in schematic form as in Figure 4, with the "grain noise". This energy is the backscatter returned to the transducer from the microstructure of the material. This signal contains information that relates to microstructure.

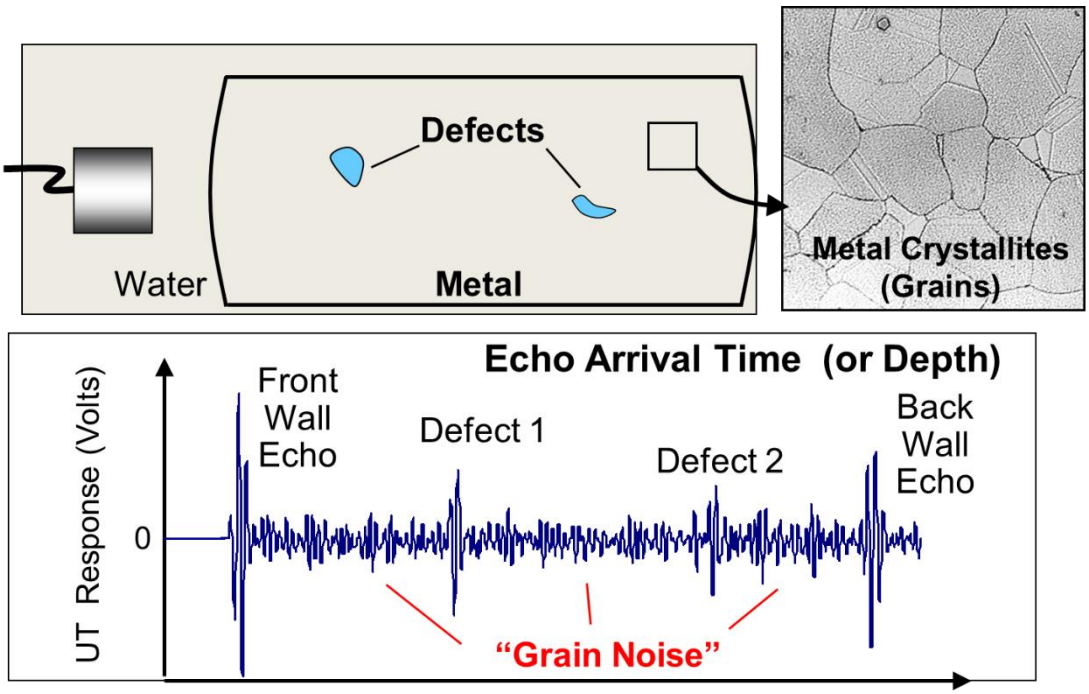


Figure 4. Diagram showing an ultrasonic inspection with backscattered grain noise.

A second modality uses the electromagnetic "Barkhausen noise " which arises from the discrete magnetic domain wall motion which occurs during cyclic magnetization. These energy bursts have been used for materials characterization [Jiles 1988]. Figure 5 shows an example of a typical magnetic Barkhausen noise signal obtained on a steel sample.

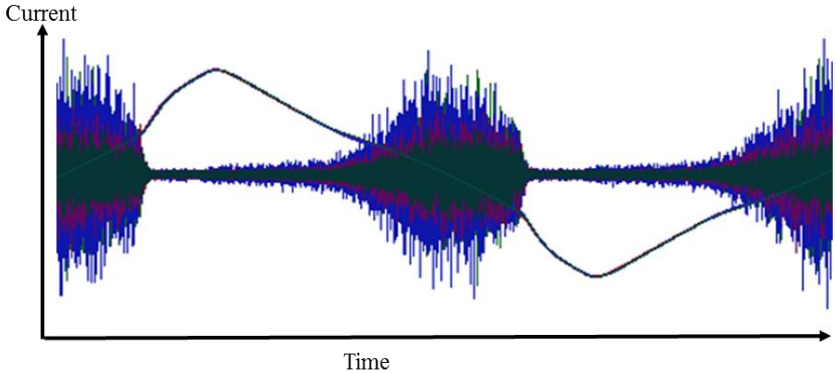


Figure 5. Example of magnetic Barkhausen signals.

3. APPROACH

There are established relationships between some NDE measurements, and in addition there are some relationships between some microstructural features and mechanical properties. The project seeks to provide the indirect relationship between NDE measurements and mechanical properties. This concept is illustrated with Figure 6.

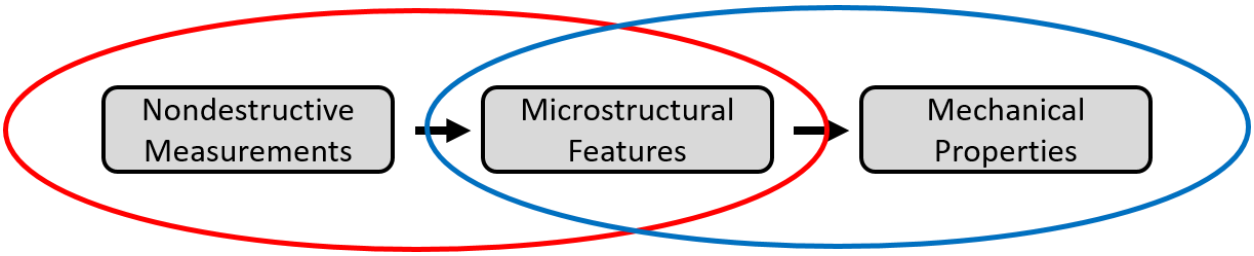


Figure 6. Project approach.

The approach to this project was to first obtain samples from industry partners, perform NDE measurements on them, and look for correlations in the results along with knowledge of mechanical properties – microstructure relationships found in the literature. As the project continued, and the lack of correlations for properties with the initial diverse sample set additional well characterized samples were sought.

The NDE measurements considered included:

- Ultrasonics
  - Velocity – speed at which the ultrasonic wave propagates; (related to elastic moduli)
  - Attenuation – measure of the amount of energy the wave lost during propagation; (related to microstructure)
  - Backscatter – measure of scattering from microstructure/grains
- Electromagnetic Barkhausen noise
  - Signals are from discrete magnetic domain motion during magnetization.

The project was divided into four tasks:

- Sample procurement
- Study the relationship between microstructure and chemistry with mechanical properties
- Study the nondestructive measurements and how they relate to the microstructure
- Assessment of the NDE approach

4. TASK 1: OBTAIN SAMPLES

4.1 Initial Pipe Samples

Five samples with varying grade were obtained from Kiefner and Associates, Inc. together with some known property data, and seven samples, five X52 and two X70, were obtained from Spectra Energy, again with some known property data.

Information about the microstructure was known for some of these samples and available data are shown in Table 1. The samples are named according to their grade, such that X52A, for example, is a sample of X52 steel, with the 'A' identifying it among other X52 samples. XGB denotes grade B steel, and XUN denotes unknown grade.

Table 1. Microstructural information for on-hand samples.

Microstructure Information				
Sample	Grain Size (ASTM #)	Avg. Grain Size (mm)	% Ferrite	Inclusion Content
X42A	12.707	0.0044	68.462	0.243
X42B				
X46A	11.557	0.0065	78.703	0.252
X52A	8	0.0225		
X52B	9	0.0159		
X52C	9	0.0159		
X52D	8.5	0.0189		
X52G	8	0.0225		
X70A				
XGBA	11.009	0.0079	71.667	0.17
XUNA				
X70B				

Two main groups of grain size can be seen in the data given Table 1. Three samples have average grain sizes of ~4-8 um, and five samples have average grain sizes of ~16-23 um. Grain size distributions are not known. Chemical composition (percent) for the initial samples is given in Table 2.

.

Table 2. Chemical composition of on-hand samples.

Chemical Composition									
Sample	C	Mn	P	S	Si	Ni	Cr	Mo	Cu
X42A	0.193	1.19	0.022	0.023	0.019	0.012	0.02	0.008	0.014
X42B	0.181	0.5	0.01	0.023	0.042	0.071	0.069	0.014	0.287
X46A	0.178	0.684	0.02	0.028	0.073	0.037	0.041	0.009	0.091
X52A	0.24	1.12	0.011	0.017	0.04	0.01	0.02	0.01	0.05
X52B	0.23	1.05	0.01	0.026	0.04	0.02	0.03	0.01	0.06
X52C	0.24	1.02	0.014	0.02	0.03	0.02	0.02	0.01	0.04
X52D	0.24	1.02	0.012	0.018	0.04	0.02	0.02	0.01	0.12
X52G	0.36	1.13	0.014	0.031	0.04	0.02	0.02	0.01	0.05
X70A	0.08	1.66	0.021	0.002	0.31	0.02	0.05	0.01	0.02
XGBA	0.254	0.557	0.006	0.041	0.044	0.039	0.019	0.005	0.049
XUNA	0.218	0.866	0.012	0.022	0.184	0.022	0.086	0.023	0.082
X70B	0.08	1.59	0.018	0.002	0.34	0.03	0.05	0.01	0.02
Chemical Composition									
Sample	V	Nb	B	Ti	Sn	Al	Zr	Ca	Co
X42A	0	0.041	0.0001	0.003	0.001	0.046	0.001	0.0002	0.001
X42B	0	0.001	0.0001	0.001	0.013	0.002	0	0.0005	0.004
X46A	0	0.001	0.0001	0.001	0.012	0.001	0	0.0003	0.007
X52A	0.008	0.008	0.005	0.008					
X52B	0.008	0.008	0.005	0.008					
X52C	0.008	0.008	0.005	0.008					
X52D	0.008	0.008	0.005	0.008					
X52G	0.008	0.008	0.005	0.008					
X70A	0.008	0.04	0.005	0.013					
XGBA	0	0.001	0.0001	0.001	0.003	0.005	0	0.0002	0.007
XUNA	0	0.001	0.0001	0.001	0.001	0.004	0	0.0003	0.002
X70B	0.008	0.039	0.005	0.015					

Destructive strength testing results for the initial samples are shown in

Table 3. This data demonstrates how the grade designation is a *minimum* specification:

- Both X42 samples have higher yield strength than the X46 samples.
- One X42 samples has higher yield strength than all but the X70 steel.
- The grade B steel has about the same yield strength as the X52 steel.

Table 3. Destructive test results for on-hand samples.

Destructive Test Results								
					Charpy Test Results			
Sample	Yield Strength (psi)	Tensile Strength (psi)	Elongation	Hardness	Test Upper-Shelf	Full-Size Upper-Shelf	Test Trans Temp.	Full-Wall Trans Temp.
X42A	70000	86000	21	187/183	8	11.9	-24	-24
X42B	56000	70000	31	159/159	15	28	179	179
X46A	51500	65500	31.8	143/146	16	32	119	119
X52A	58000	79200	35					
X52B	57500	78600	35					
X52C	56400	79200	30					
X52D	54900	75800	34.5					
X52G	61500	87900	30					
X70A	75500	90700	34.5					
XGBA	58000	74000	25.6	152/156	13	35	116	120
XUNA	47400	69500	37.5	170/167	32	43	135	135
X70B	80000	92200	34					

The set of samples has what seems to be inconsistent microstructural/destructive known properties. The ranges and potential uncertainty/ errors in the stated values are not known.

4.2 Kiefner Sample Database

An alternate source of pipeline sample data was obtained from Kiefner and the data provided consisted of chemistry, microstructure, and strength information for dozens of pipeline samples. The physical samples were not available for nondestructive measurements but these data had value for relating the microstructure and chemistry information to the strength properties.

The data for samples included in the data base was reviewed and Figure 7 shows several histograms reporting the distributions found in some sample metrics. It can be seen that X52 is very common in this sample set and that there is a very large distribution in terms of the vintage of the pipe. The age distribution is important as it is know that there have been significant changes in the manufacturing processes over time [Gray and Siciliano, 2009].

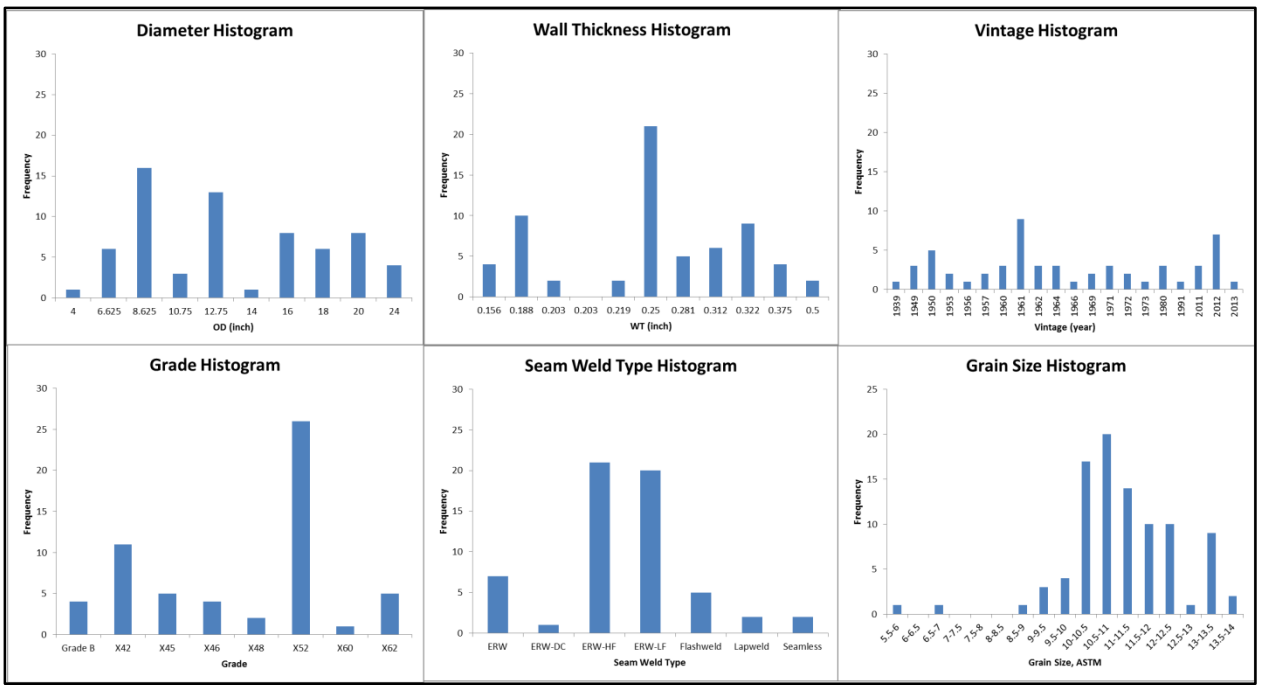


Figure 7. Histograms for distribution of properties reported in the Kiefner sample database.

The variability of strength properties of these for these samples can be seen plotted as functions of the microstructural properties, as Figure 8 and Figure 9. Figure 8 is color-coded according to vintage (for those samples with known vintage) and Figure 9 color-coded according to grade (for those samples with known grade). Looking at the complete sample set, the properties are found to be tightly grouped. In reviewing these data a few trends can be seen:

- The relationship seen between yield strength and grain size loosely follows the Hall-Petch relationship, which says the yield strength is linearly related to the inverse square root of the grain size. [*Note that Figure 8 and Figure 9 show the grain size rather than the inverse square root of grain size so the relationship does not look linear*].

- There is a trend seen that shows that yield and tensile strength increase with increasing manganese content.

- The data in Figure 8 highlights the fact that in general newer pipeline steels have higher strength but are lower in carbon. When disregarding the newest pipe samples (>2000) a trend can be seen to exist between tensile strength and carbon content.

- A weak relationship is seen to be present between percent ferrite and tensile strength.

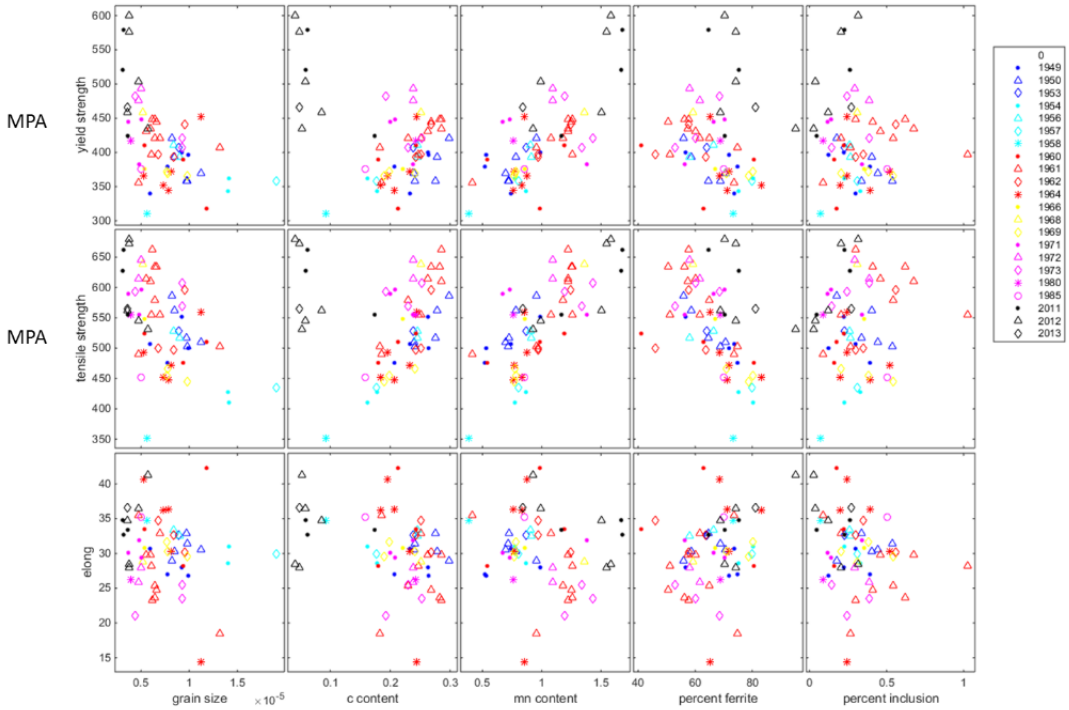


Figure 8. Data base sample parameters color-coded with vintage.

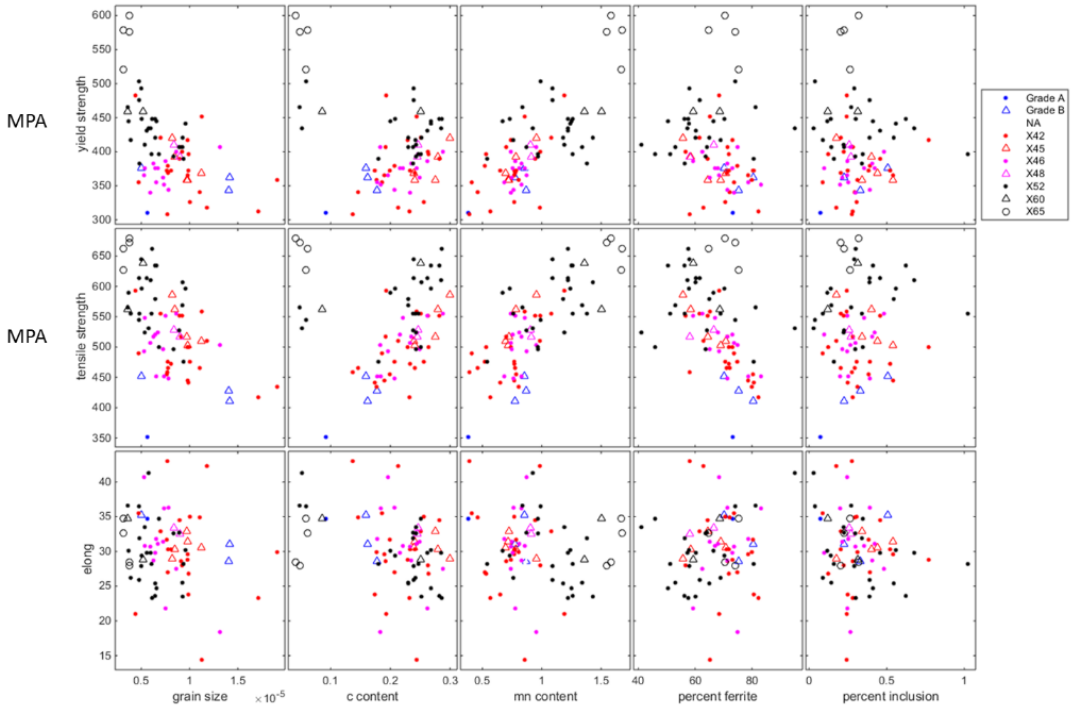


Figure 9. Data base sample parameters color-coded with grade.

5. TASK 2: RELATIONSHIP BETWEEN MATERIAL AND MECHANICAL PROPERTIES

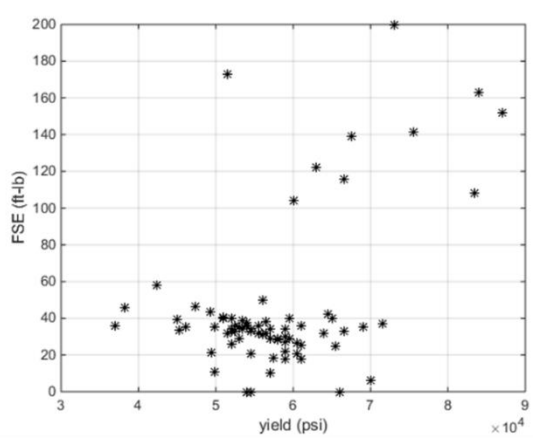
Task 2 considered the relationships between the mechanical properties of the steels and the other known material properties of the steels, such as the microstructure and chemical composition. Several relationships were found reported in the surveyed literature that were expected to be seen when the data in the data base was reviewed. What was found, however, was that the relationships reported as conventional wisdom as the relationships between mechanical and material properties do not hold well for the data on the samples in the data base that was available for this study.

This is a key problem which was not expected to be seen and that was not within scope of this project. In defining this project it had been assumed that if the microstructure and chemical composition of the steels could be obtained nondestructively, that information then could in turn be used to estimate the mechanical properties of the steels. This is a materials and statistics issue, with relation to seeking correlations between pipe material samples and pipe strength parameters which will need to be better understood before correlations between NDE data and material properties can be used to predict strength.

To highlight this issue an excerpt from Samuels [1999] which mentioned several generally assumed relationships for carbon steels is reproduced and discussed together with examples of data from the data base.

The first relationship highlighted is that between toughness and yield strength. Samuels states that, in general, toughness decreases as yield strength is raised. Figure 10 shows that this is not seen in the available sample data.

The general problem is that fracture toughness in conventional steels decreases roughly linearly as yield strength is raised, although the relationship between the two is significantly different for different types of steel. The principal factor affecting tensile and yield strengths is carbon content, which influences tensile strength mainly through increasing the amount of pearlite present. Increasing the volume of pearlite also has the direct effect of causing a significant deterioration in fracture toughness. The direct effect of pearlite on yield strength, however, is negligible, but there is an indirect effect in that increasing the volume of pearlite tends to reduce ferritic grain size by blocking growth of the ferrite grains. Reducing ferritic grain size by this or any other means increases yield strength, which varies approximately linearly with the reciprocal of the square root of the grain diameter of the ferrite. Reducing the ferritic grain size also beneficially affects fracture toughness; it is the one factor that improves yield strength and toughness simultaneously. Alloying elements other than carbon have only small direct effects on strength due to solid-solution hardening; they may produce indirect effects by influencing the volume of pearlite present, by affecting the ferrite grain size or, as we shall see later, by introducing precipitation effects.



Samuels, Leonard Ernest. *Light Microscopy of Carbon Steels*. ASM International, 1999.

Figure 10. Yield strength – toughness relationship – showing parameters for typical data base samples.

However, the cluster pattern seen in the data shown in Figure 10 may indicate that there is another variable affecting the results in parameters for this sample set. When reanalyzing the data base for vintage, removing pipe newer than 1980, there is a found to be a slight negative linear relationship present (Figure 11). There is one outlying data point, that for a pipe from 1961. This is an indication that pipe vintage may be important to consider when determining material relationships. The expected relationship stated by Samuels do not hold for pipe newer than 1980.

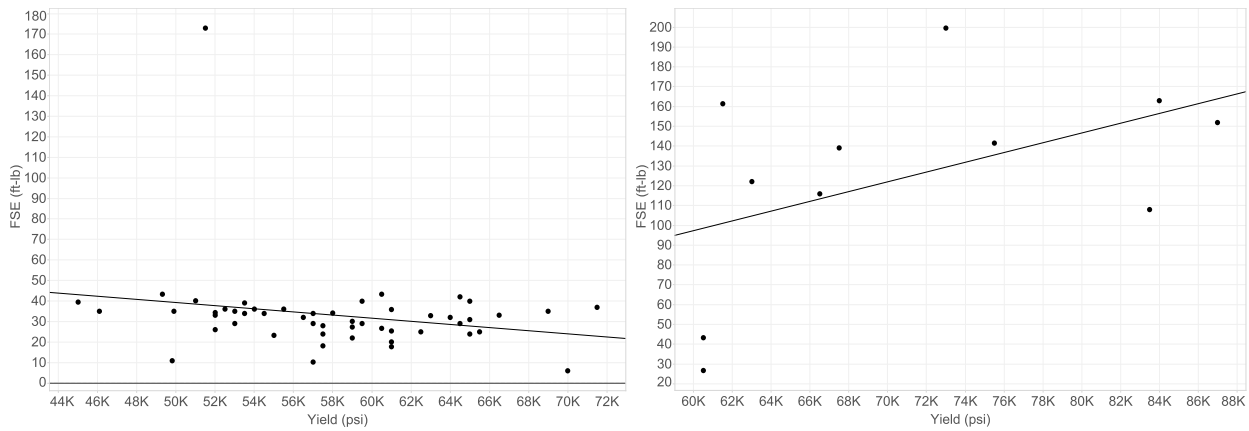
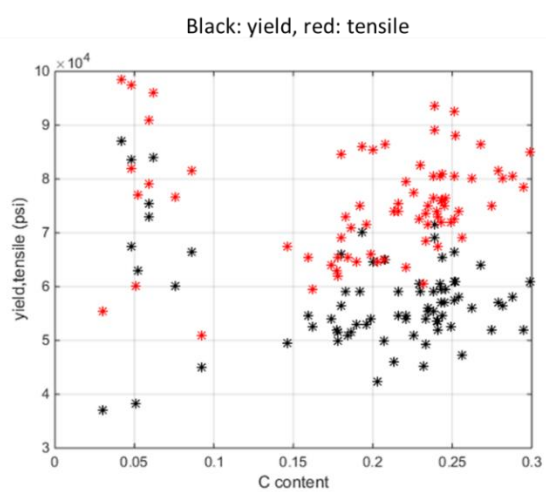


Figure 11. Yield strength - toughness relationship, (a) pipe older than 1980, (b) pipe newer than 1980.

Samuels states that carbon content is the principal factor affecting tensile and yield strength. Figure 12 shows the relationship between carbon content and yield and tensile strength. It can be seen that there is a grouping of low carbon, high strength steels, which were noted earlier to be recently manufactured (>2000) steels. Disregarding these, a slight trend can be seen between carbon content and tensile strength, so data for these samples are agreeing with Samuels.

The general problem is that fracture toughness in conventional steels decreases roughly linearly as yield strength is raised, although the relationship between the two is significantly different for different types of steel. The principal factor affecting tensile and yield strengths is carbon content, which influences tensile strength mainly through increasing the amount of pearlite present. Increasing the volume of pearlite also has the direct effect of causing a significant deterioration in fracture toughness. The direct effect of pearlite on yield strength, however, is negligible, but there is an indirect effect in that increasing the volume of pearlite tends to reduce ferritic grain size by blocking growth of the ferrite grains. Reducing ferritic grain size by this or any other means increases yield strength, which varies approximately linearly with the reciprocal of the square root of the grain diameter of the ferrite. Reducing the ferritic grain size also beneficially affects fracture toughness; it is the one factor that improves yield strength and toughness simultaneously. Alloying elements other than carbon have only small direct effects on strength due to solid-solution hardening; they may produce indirect effects by influencing the volume of pearlite present, by affecting the ferrite grain size or, as we shall see later, by introducing precipitation effects.



Samuels, Leonard Ernest. *Light Microscopy of Carbon Steels*. ASM International, 1999.

Figure 12. Strength – carbon content relationship.

The relationship between multiple chemical contents and yield strength is shown in Figure 13. These plots have a color-coded scale for the age of the pipe, blue indicating newer pipe, red is older. Regardless of vintage, the manganese content shows a strong relationship with yield strength. However, in all plots, there is a tendency for the newer pipe to follow relationships between parameters which are different from those for the older pipe.

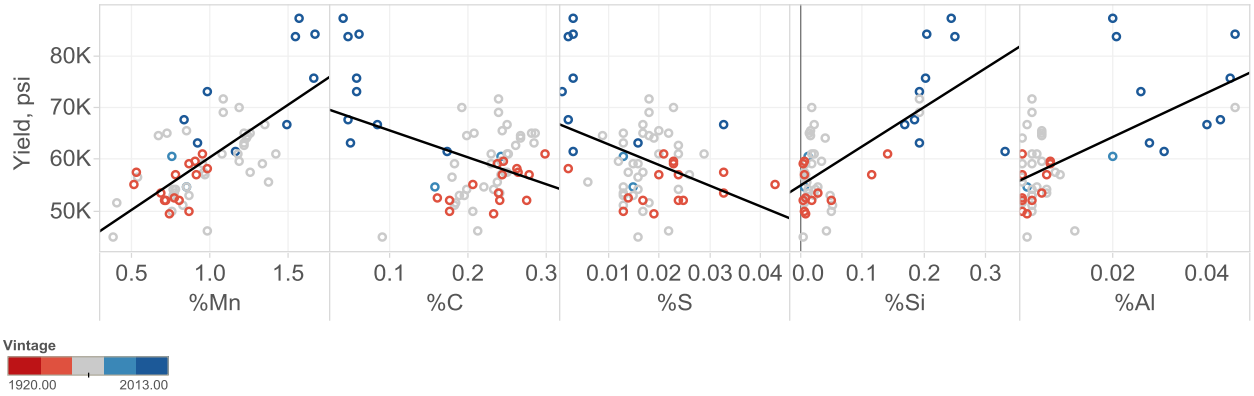
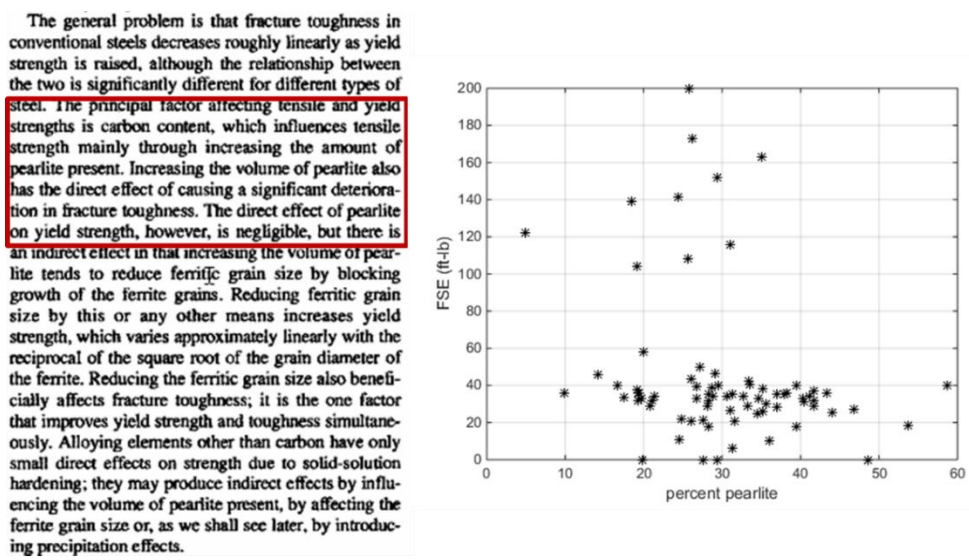


Figure 13. Yield strength - chemical content relationship.

The data for the relationship between the full-shelf energy (FSE) from the Charpy test as a metric of toughness with the percent pearlite of the microstructure is given in Figure 14. These data relate to the second part of the statement highlighted in Figure 112. The trend Samuels expected from is not seen for the data base samples for the relationship between these two variables.



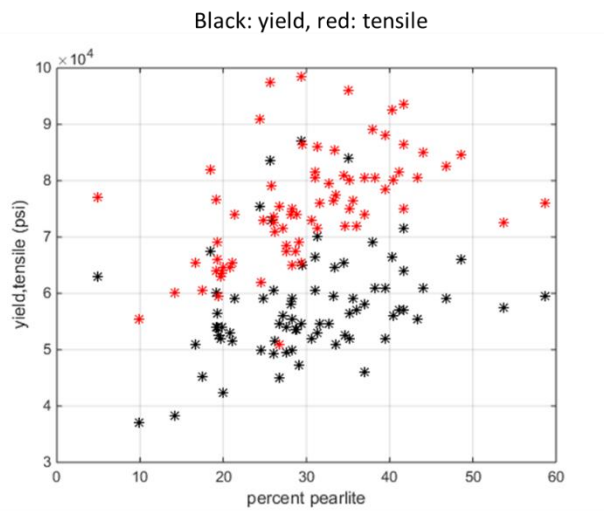
The general problem is that fracture toughness in conventional steels decreases roughly linearly as yield strength is raised, although the relationship between the two is significantly different for different types of steel. The principal factor affecting tensile and yield strengths is carbon content, which influences tensile strength mainly through increasing the amount of pearlite present. Increasing the volume of pearlite also has the direct effect of causing a significant deterioration in fracture toughness. The direct effect of pearlite on yield strength, however, is negligible, but there is an indirect effect in that increasing the volume of pearlite tends to reduce ferritic grain size by blocking growth of the ferrite grains. Reducing ferritic grain size by this or any other means increases yield strength, which varies approximately linearly with the reciprocal of the square root of the grain diameter of the ferrite. Reducing the ferritic grain size also beneficially affects fracture toughness; it is the one factor that improves yield strength and toughness simultaneously. Alloying elements other than carbon have only small direct effects on strength due to solid-solution hardening; they may produce indirect effects by influencing the volume of pearlite present, by affecting the ferrite grain size or, as we shall see later, by introducing precipitation effects.

Samuels, Leonard Ernest. *Light Microscopy of Carbon Steels*. ASM International, 1999.

Figure 14. Toughness – percent pearlite relationship.

The relationship between strength and percent pearlite is shown in Figure 15. Samuels states that the effect of percent pearlite on yield strength is negligible. However, a slight upward trends can be seen in these data for the relationship between both yield and tensile strength with pearlite content.

The general problem is that fracture toughness in conventional steels decreases roughly linearly as yield strength is raised, although the relationship between the two is significantly different for different types of steel. The principal factor affecting tensile and yield strengths is carbon content, which influences tensile strength mainly through increasing the amount of pearlite present. Increasing the volume of pearlite also has the direct effect of causing a significant deterioration in fracture toughness. The direct effect of pearlite on yield strength, however, is negligible, but there is an indirect effect in that increasing the volume of pearlite tends to reduce ferritic grain size by blocking growth of the ferrite grains. Reducing ferritic grain size by this or any other means increases yield strength, which varies approximately linearly with the reciprocal of the square root of the grain diameter of the ferrite. Reducing the ferritic grain size also beneficially affects fracture toughness; it is the one factor that improves yield strength and toughness simultaneously. Alloying elements other than carbon have only small direct effects on strength due to solid-solution hardening; they may produce indirect effects by influencing the volume of pearlite present, by affecting the ferrite grain size or, as we shall see later, by introducing precipitation effects.

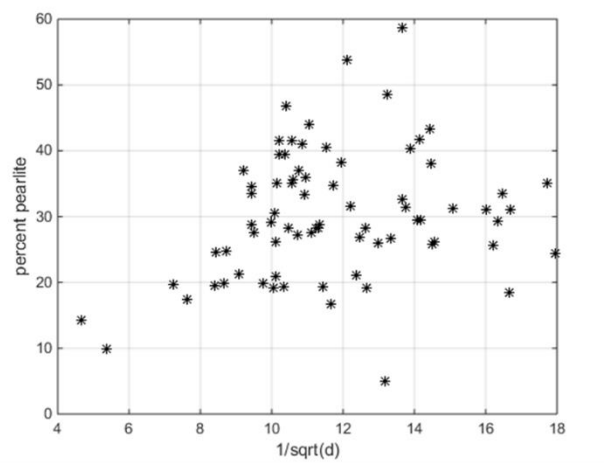


Samuels, Leonard Ernest. *Light Microscopy of Carbon Steels*. ASM International, 1999.

Figure 15. Strength – percent pearlite relationship.

The relationship between percent pearlite and the inverse square root of grain size is shown in Figure 16. Samuels states that an increase in pearlite content tends to reduce ferritic grain size. The expected trend is somewhat present for large grains, but there is a significant scatter in the data, which is particularly so for smaller grains (inverse grain sizes greater than 10 $mm^{-0.5}$).

The general problem is that fracture toughness in conventional steels decreases roughly linearly as yield strength is raised, although the relationship between the two is significantly different for different types of steel. The principal factor affecting tensile and yield strengths is carbon content, which influences tensile strength mainly through increasing the amount of pearlite present. Increasing the volume of pearlite also has the direct effect of causing a significant deterioration in fracture toughness. The direct effect of pearlite on yield strength, however, is negligible, but there is an indirect effect in that increasing the volume of pearlite tends to reduce ferritic grain size by blocking growth of the ferrite grains. Reducing ferritic grain size by this or any other means increases yield strength, which varies approximately linearly with the reciprocal of the square root of the grain diameter of the ferrite. Reducing the ferritic grain size also beneficially affects fracture toughness; it is the one factor that improves yield strength and toughness simultaneously. Alloying elements other than carbon have only small direct effects on strength due to solid-solution hardening; they may produce indirect effects by influencing the volume of pearlite present, by affecting the ferrite grain size or, as we shall see later, by introducing precipitation effects.

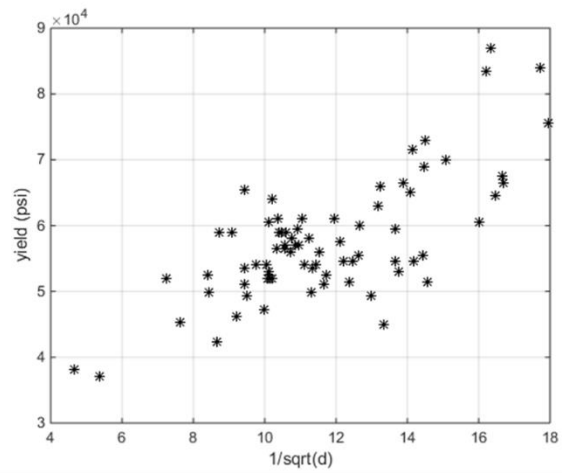


Samuels, Leonard Ernest. *Light Microscopy of Carbon Steels*. ASM International, 1999.

Figure 16. Percent pearlite – grain size relationship.

The relationship between grain size and yield strength is shown in Figure 17. Samuels states that yield strength varies approximately linearly with the inverse square root of the grain size. This trend is the most clearly seen of all that are mentioned here, but it should be noted that for a given grain size there is still a variation of up to 20,000 psi or more in the yield strength. This is an important point which highlights the challenges encountered with this project. If this relationship between grain size and yield strength, which is the best trend seen in the data, were to be used to predict the yield strength from the grain size, it would not give very accurate estimates for the yield strength. Even if the grain size could be predicted with minimal error (uncertainty) nondestructively there is still a significant range or variation in a prediction.

The general problem is that fracture toughness in conventional steels decreases roughly linearly as yield strength is raised, although the relationship between the two is significantly different for different types of steel. The principal factor affecting tensile and yield strengths is carbon content, which influences tensile strength mainly through increasing the amount of pearlite present. Increasing the volume of pearlite also has the direct effect of causing a significant deterioration in fracture toughness. The direct effect of pearlite on yield strength, however, is negligible, but there is an indirect effect in that increasing the volume of pearlite tends to reduce ferritic grain size by blocking growth of the ferrite grains. Reducing ferritic grain size by this or any other means increases yield strength, which varies approximately linearly with the reciprocal of the square root of the grain diameter of the ferrite. Reducing the ferritic grain size also beneficially affects fracture toughness; it is the one factor that improves yield strength and toughness simultaneously. Alloying elements other than carbon have only small direct effects on strength due to solid-solution hardening; they may produce indirect effects by influencing the volume of pearlite present, by affecting the ferrite grain size or, as we shall see later, by introducing precipitation effects.

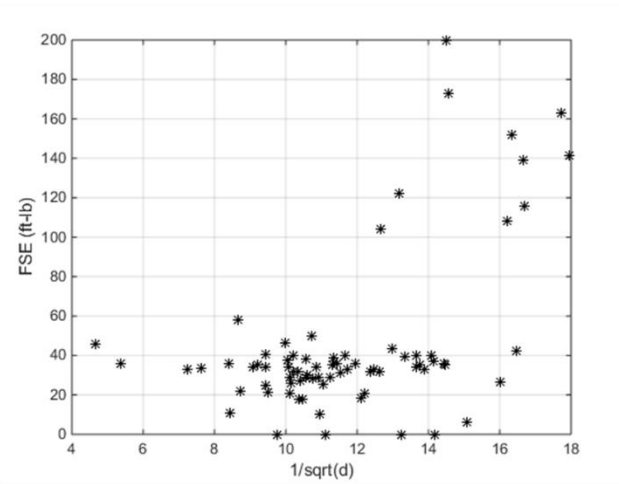


Samuels, Leonard Ernest. *Light Microscopy of Carbon Steels*. ASM International, 1999.

Figure 17. Yield strength – grain size relationship.

The relationship between grain size and a toughness metric, the full-shelf energy from a Charpy test is shown in Figure 18. No consistent trend is seen, but the samples with high toughness tend to have smaller grains, which agrees with Samuels.

The general problem is that fracture toughness in conventional steels decreases roughly linearly as yield strength is raised, although the relationship between the two is significantly different for different types of steel. The principal factor affecting tensile and yield strengths is carbon content, which influences tensile strength mainly through increasing the amount of pearlite present. Increasing the volume of pearlite also has the direct effect of causing a significant deterioration in fracture toughness. The direct effect of pearlite on yield strength, however, is negligible, but there is an indirect effect in that increasing the volume of pearlite tends to reduce ferritic grain size by blocking growth of the ferrite grains. Reducing ferritic grain size by this or any other means increases yield strength, which varies approximately linearly with the reciprocal of the square root of the grain diameter of the ferrite. Reducing the ferritic grain size also beneficially affects fracture toughness; it is the one factor that improves yield strength and toughness simultaneously. Alloying elements other than carbon have only small direct effects on strength due to solid-solution hardening; they may produce indirect effects by influencing the volume of pearlite present, by affecting the ferrite grain size or, as we shall see later, by introducing precipitation effects.



Samuels, Leonard Ernest. *Light Microscopy of Carbon Steels*. ASM International, 1999.

Figure 18. Toughness – grain size relationship.

5.1 Linear Modeling: Mechanical/Material Property Relationships

In an effort to address the issue of relating mechanical properties to material properties some linear modeling was developed. The starting point of the modeling was assuming that the yield strength is linearly dependent on the hardness of the steel. Additional variables were added in groups to see the effects they have on the modeling results. Four models were assessed for yield strength:

- yield as a function of hardness
- yield as a function of hardness and microstructure (grain size, percent pearlite, percent inclusion as independent variables)
- yield as a function of hardness, microstructure, and the carbon equivalent, which is a weldability parameter
- yield as a function of hardness, microstructure, and individual chemical elements.

The modeling was performed using linear regression and the parameters for the database samples. The parameters for the various samples were then run through the models to determine an estimate of the yield strength. The results of this modeling are given as histograms showing how many of the samples from the database give a predicted yield strength within a certain percent of the actual (experimental) yield strength. These results are shown in Figure 19. It should be noted that the bins denoting $\pm$10-20% error actually include all errors beyond $\pm$20%.

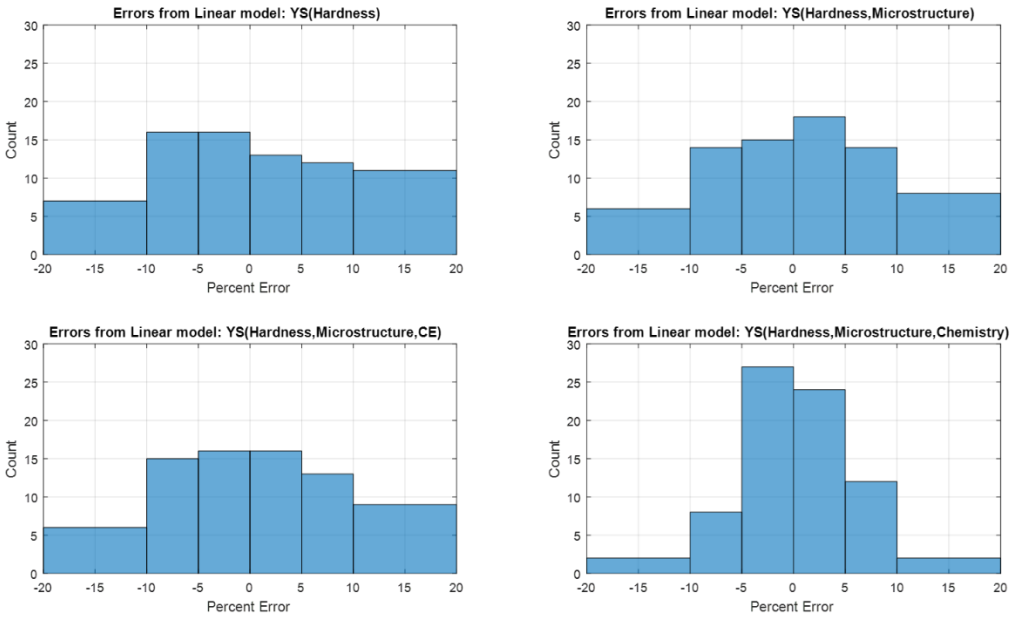


Figure 19. Linear modeling of yield strength. Clockwise from top left: a.) yield as a function of hardness, b.) yield as a function of hardness and microstructure, c.) yield as a function of hardness, microstructure, and carbon equivalent, d.) yield as a function of hardness, microstructure, and each chemical element.

It can be seen in Figure 19 that incremental improvements in yield strength prediction occur when additional variables are added, leading to the best prediction when yield strength is modeled as a function of hardness, microstructure, and the individual chemical elements. The linear model using just hardness gives 57 out of 75 samples having yield strength within 10% of the actual value. Adding microstructure gives 61 out of 75 having less than 10% error, while adding microstructure and carbon equivalent gives 60 out of 75 samples with less than 10% error. Including each chemical element as an independent variable gives 71 out of 75 samples having a predicted yield strength within 10% of the actual value.

If the t-statistics for each model are plotted, the contribution to the yield strength prediction can be seen. This data is shown in Figure 20.

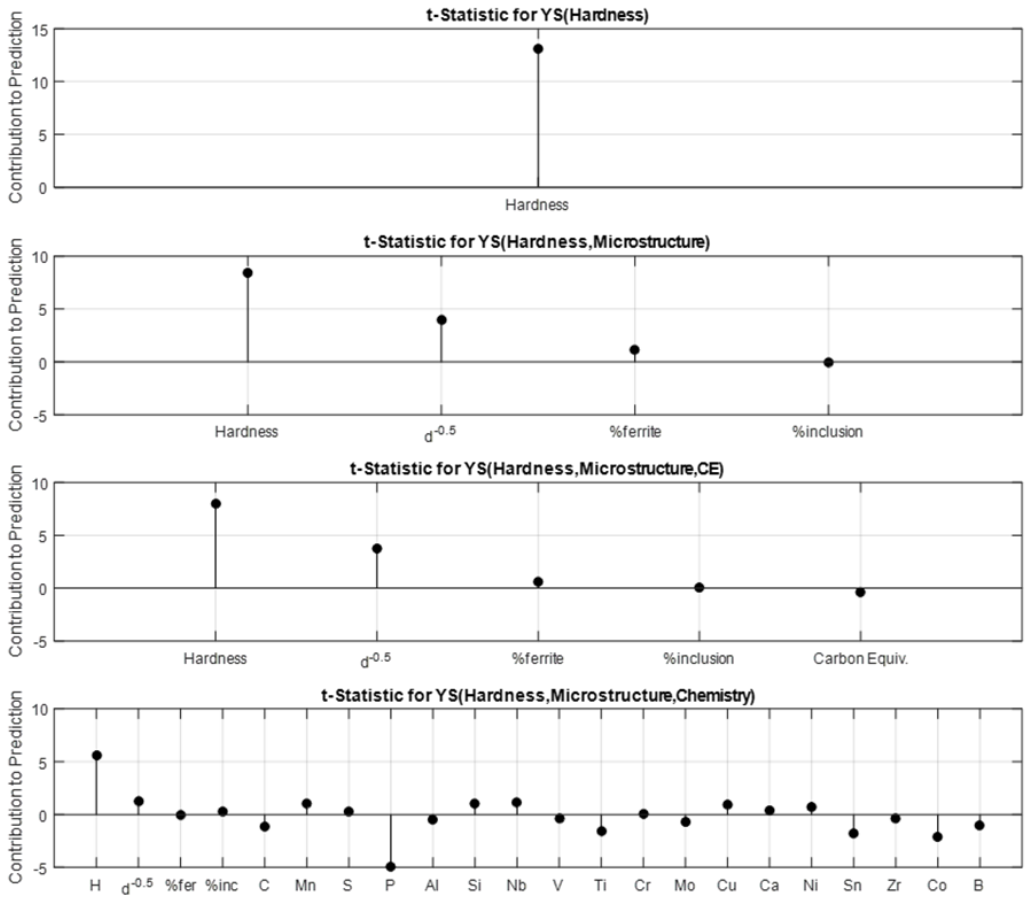


Figure 20. t-statistics, or contributions to prediction, for each model. From top to bottom: a.) yield as a function of hardness, b.) yield as a function of hardness and microstructure, c.) yield as a function of hardness, microstructure, and carbon equivalent, d.) yield as a function of hardness, microstructure, and each chemical element.

The top three plots show that the hardness and grain size are the key predictors for their respective models. The bottom plot adds the contributions to the yield strength prediction for all of the chemical elements, which gives some interesting results. These results indicate that phosphorus has almost as much influence on yield strength as hardness, which is a surprising result. Another noteworthy result is that carbon content is given to have a negative impact on yield; said another way, increasing carbon content decreases yield strength according to these results. This goes against the rule of thumb noted by Samuels but could be influenced by the newer low-carbon high-strength steels. Phosphorus is also reported as having a negative impact on yield strength, which goes against what Krauss [2005] reports. The percent ferrite and percent inclusion of the microstructure have less impact than most of the chemical elements according to this model, and grain size has a smaller effect than a number of elements. It was anticipated that the microstructure would have a larger impact on the yield strength prediction. One hypothesis that might explain these results is that the chemical elements are indicative of the manufacturing history of the steel, which is not obvious at first glance but shows up statistically. It is recommended that a more in-depth study is performed to investigate this topic.

The linear modeling results for four mechanical properties (yield strength, tensile strength, toughness, and transition temperature) are shown below in Figure 21 through 24. Each figure contains of two plots: one showing the predicted value vs. the actual value, and the second is a histogram, which is like that shown previously, containing both the model using just hardness and the model using hardness, microstructure, and each chemical element as an independent variable. Note that when the two histograms are overlaid it is denoted by a color change.

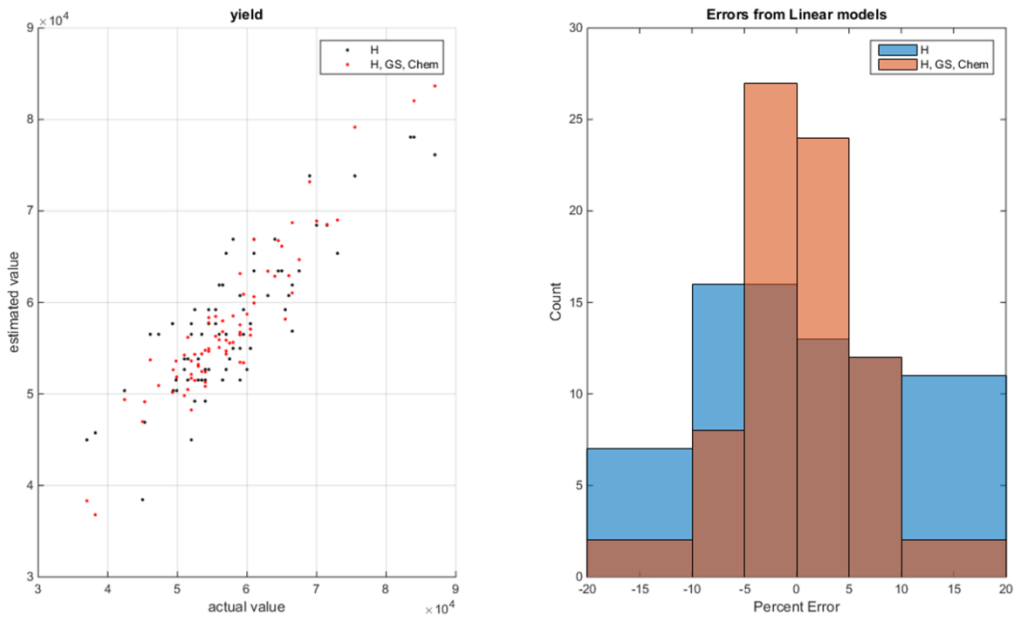


Figure 21. Linear modeling of yield strength.

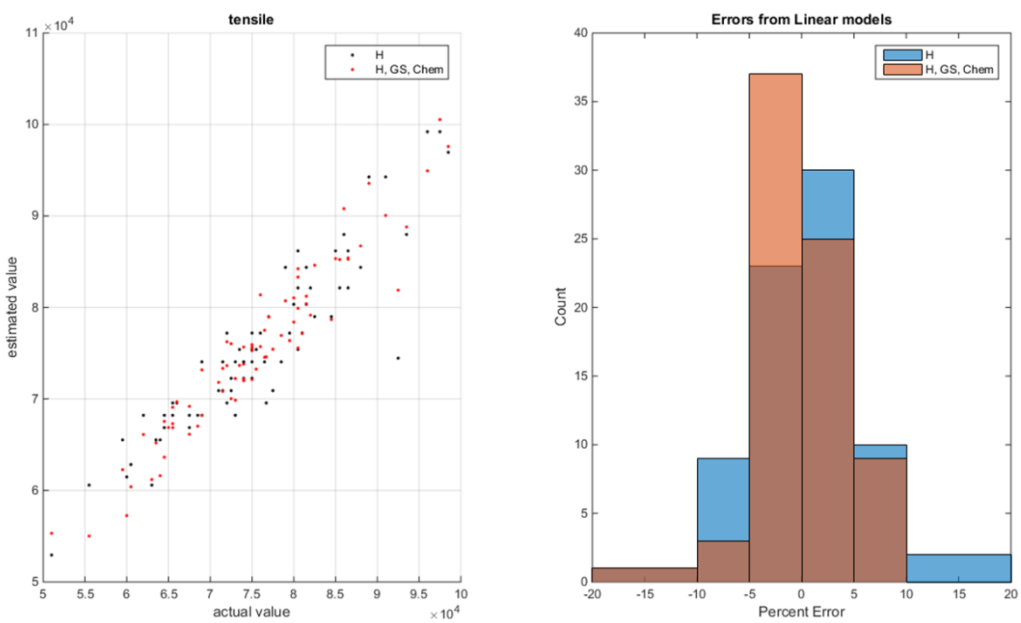


Figure 22. Linear modeling of tensile strength.

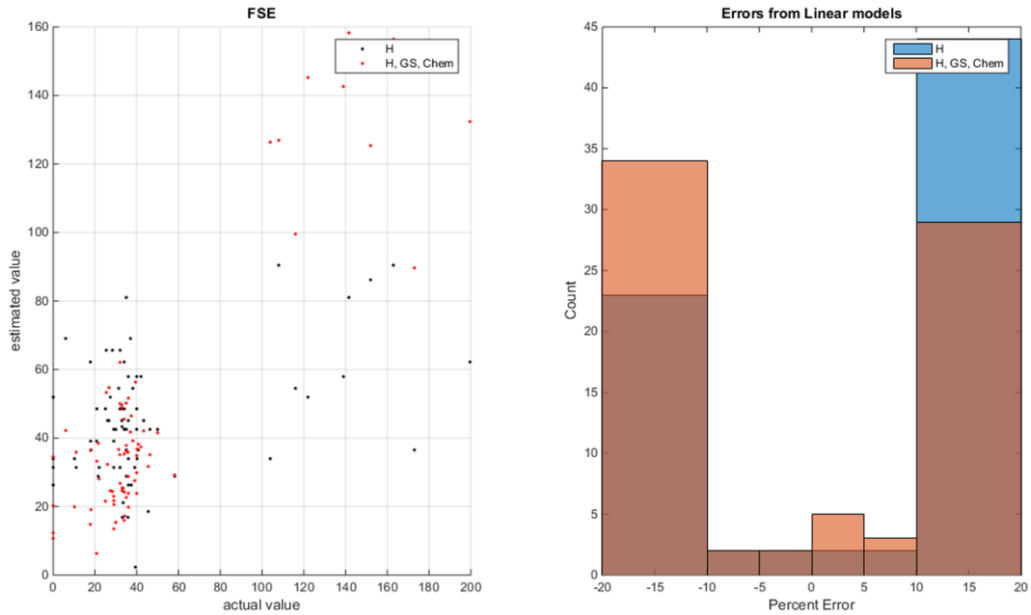


Figure 23. Linear modeling of Charpy full-shelf energy.

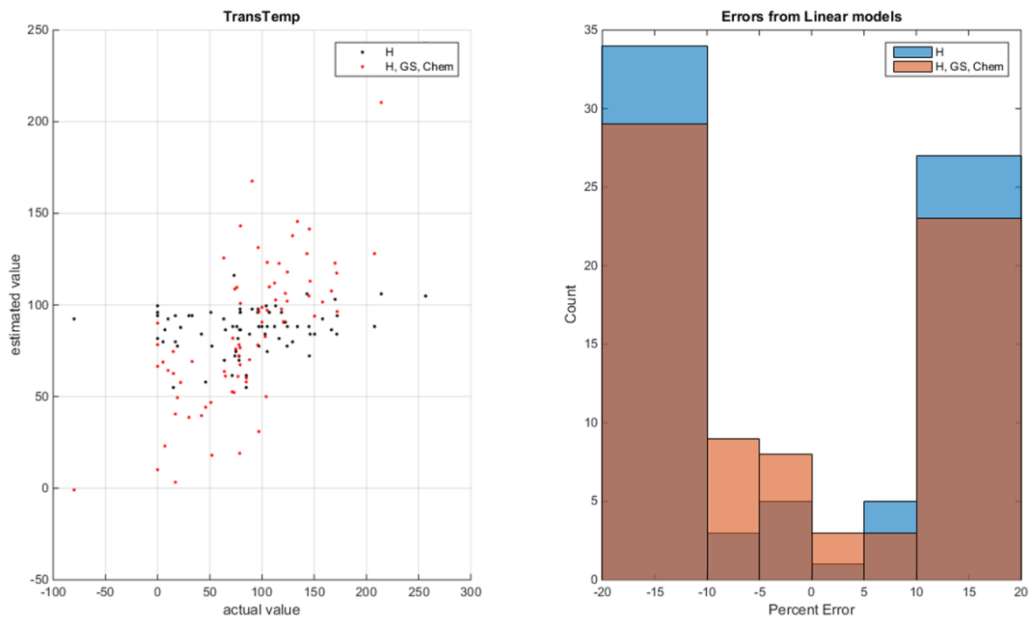


Figure 24. Linear modeling of ductile to brittle transition temperature.

It can be seen from Figure 21 through 24 that the linear models for prediction of yield and tensile strength result in most samples having estimated strength values within ±10% when hardness, microstructure, and each chemical element are taken into account. This is not the case for toughness and transition temperature, where a majority of the model-predicted values are outside the ±10% error bands. There is clearly a need for a better understanding of how the microstructure of these steels relates to the toughness and transition temperature.

6. TASK 3: NONDESTRUCTIVE MEASUREMENTS

6.1 Pipe Samples

Multiple rounds of nondestructive measurements were performed on some or all of the physical hand samples at CNDE. The first round looked at the ultrasonic velocity, attenuation, and backscatter for three samples: XGBA (A), X42A (B), X46A (C). The measurements were performed at multiple locations per sample. The methods used are those reported by Margetan et al. [2014].

The velocity results are shown in Figure 25. The variation in velocity seen within each sample is larger than the difference between the sample means. This variability is also reflected in the attenuation and backscatter results that are shown in Figures 26 and 27, respectively.

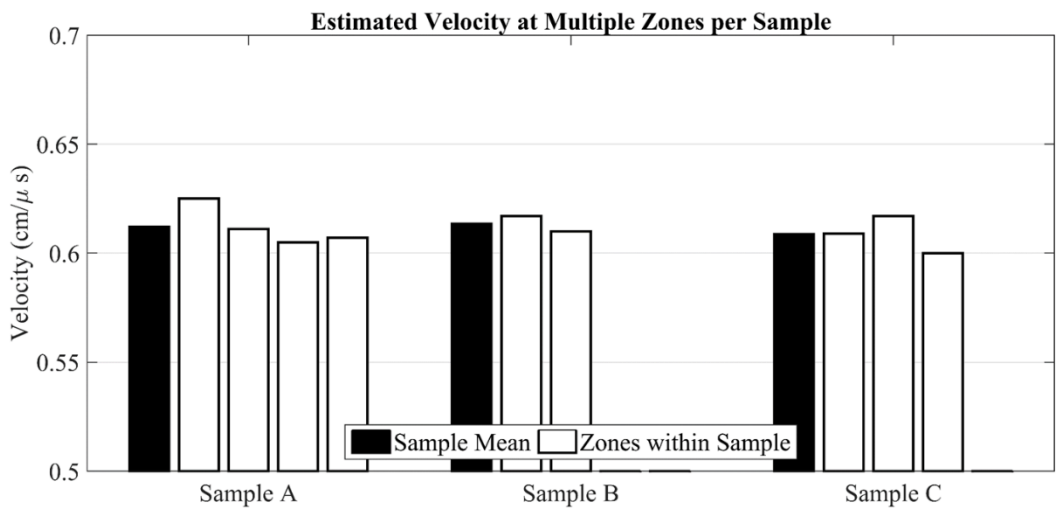


Figure 25. Velocity results for three samples.

The attenuation results are shown in Figure 26. There is a significant variability `seen in each sample, especially sample X42A, denoted as B here. This sample had axial curvature which may have prevented consistent back wall signals from being obtained, leading to artificially high attenuation estimates.

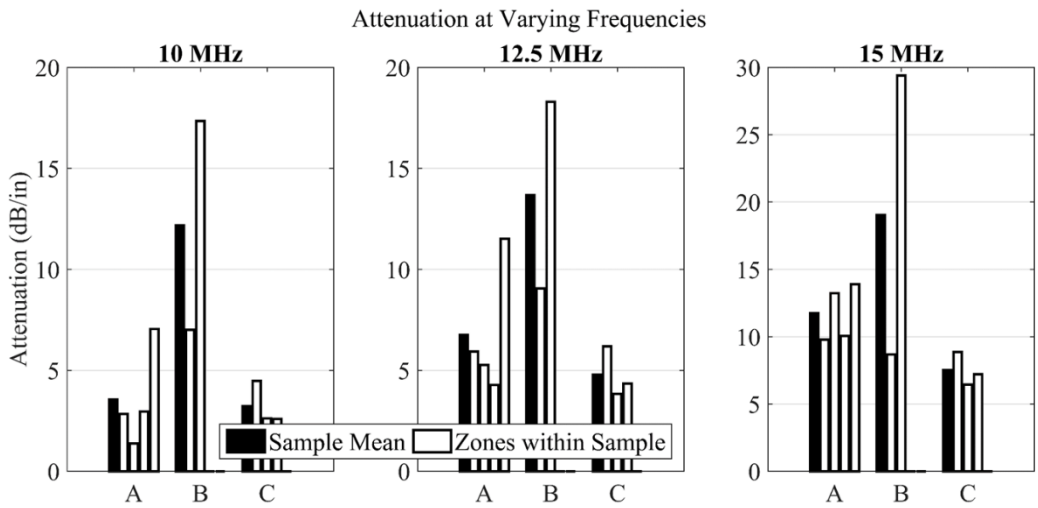


Figure 26. Attenuation results for three samples at three frequencies.

The results for the ultrasonic backscatter are shown in Figure 27. Again, large variation could be seen within each sample. This could be due to the thin walls of these samples, which can lead to the front wall signal masking the backscatter signal.

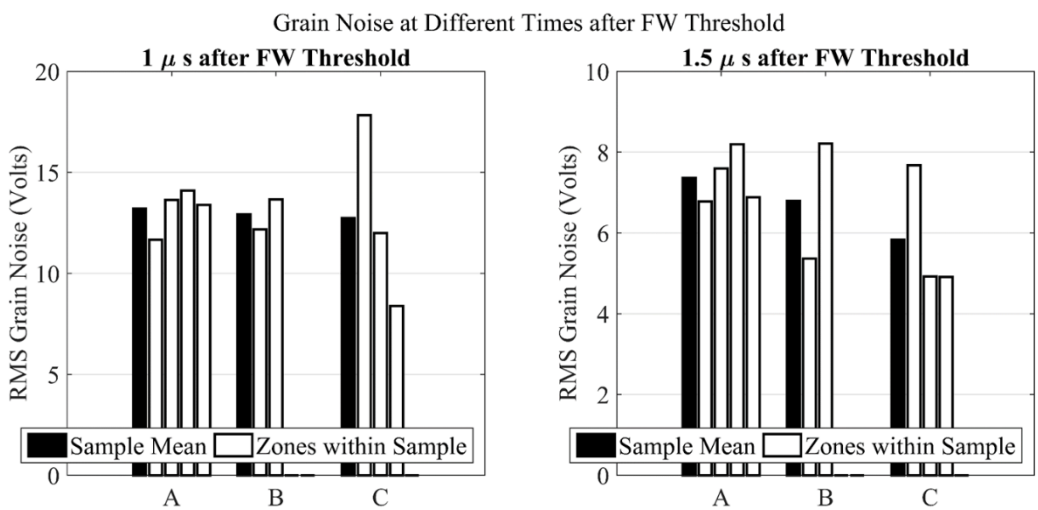


Figure 27. Backscatter results for three samples at two times.

This round of measurements proved inconclusive, and continued to be so when more samples were tested. Figure 28 shows attenuation results for all physical samples together with measured grain size and yield strength. It was expected that there would be correlations between both attenuation and grain size as well as grain size and yield strength, but neither were seen in the measured data.

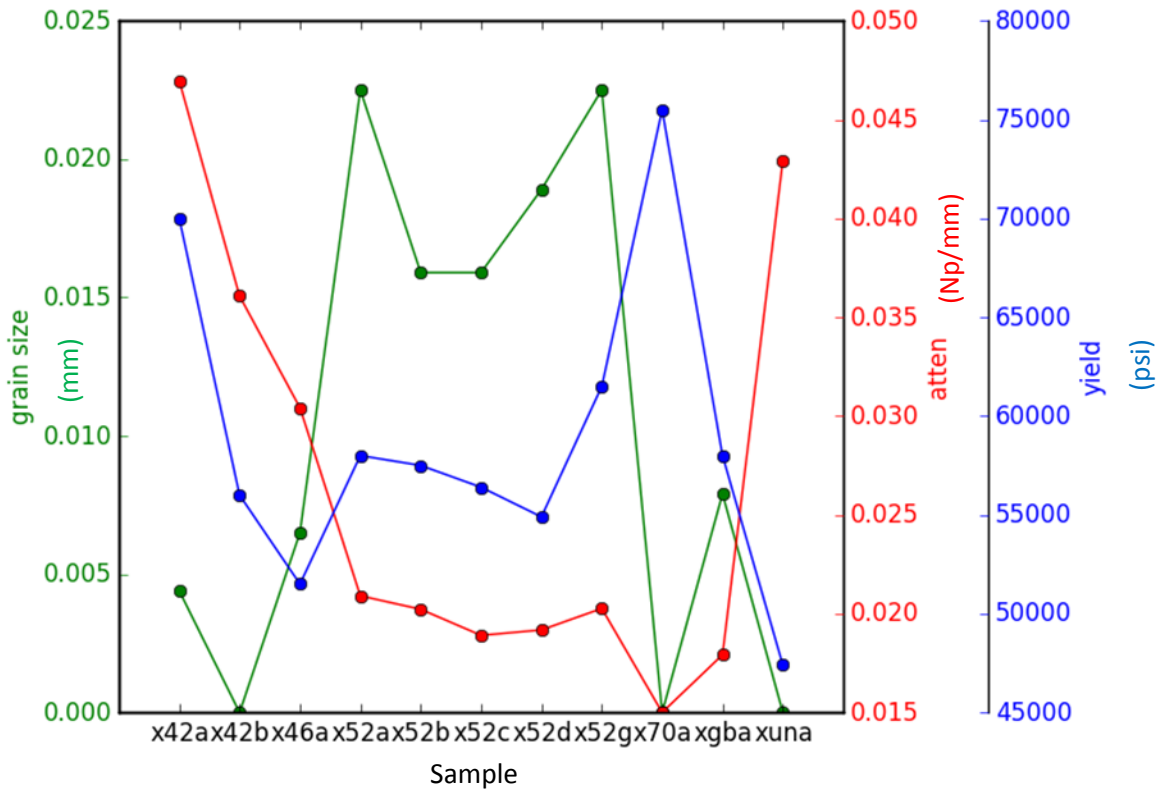


Figure 28. Grain size, ultrasonic attenuation, and yield strength for the on-hand samples.

The inconclusiveness of the nondestructive measurements highlighted a major issue with the initial sample set: there is a very large variation of steels being tested. There are multiple variables that could be changing between samples, including manufacturing process parameters, chemistry and vintage that all cause changes in grain size, pearlite content, inter-lamellar spacing in the form of the pearlite, and inclusion type and content. The various microstructural and compositional variables all make contributions (of varying significance) in the data obtained using the nondestructive measurement methods. These relationships are complex and with the available samples and data base parameters the various correlations and connections quite simply could not be determined. Variability has been seen in the measured NDE data. However, understanding these connections between NDT data and microstructure and microstructure and strength need to be determined before it will be possible to connect NDT data to strength parameters.

6.2 Controlled Samples

As an initially step towards seeking to solve the strength – NDE data relationship it was clear that it was necessary to obtain a new class of samples. Samples extracted from pipe or pipelines quite simply had too much variability to enable the multi-variant problem to be unraveled.

Various organizations were contacted to seek to obtain well qualified samples (i.e. NIST, Battelle and Colorado School of Mines). As an initial step to give well controlled samples it was

decided to investigate the effects of microstructural changes using a set of X52 steel samples which were obtained from Colorado School of Mines (CSM). CSM has a pilot scale mill that was used to give samples exposed to different heat treatments. The samples were then studied using the various nondestructive techniques. Unlike the previously mentioned samples, these were not from sections of actual pipelines. The sample set consisted of six samples of approximately 2”x 2”, and 0.5” thickness steel plate:

- Sample A: control; no heat treatment
- Sample B: normalized at 950C for one hour, air cooled
- Sample C: normalized at 950C for one hour, furnace cooled
- Sample D: Annealed at inter-critical temperature of 800C, air cooled
- Sample E: Annealed at inter-critical temperature of 800C, furnace cooled
- Sample F: Heated to 1150C, furnace cooled for two hours
-

Samples A-E mainly had varying volume fractions of ferrite and pearlite. Sample F was created in an attempt to greatly increase the grain size. Parameters are shown in Table 4.

Table 4. Microstructure of controlled samples.

Sample	Volume Fraction Ferrite	Volume Fraction Pearlite	Grain Size (um)
A	0.939	0.061	11.8
B	0.856	0.144	11.3
	0.859	0.141	10.4
C	0.89	0.11	10.9
	0.897	0.103	10
D	0.826	0.174	10.5
	0.804	0.196	10.5
E	0.8	0.2	11.5
	0.852	0.148	11.2
F	0.905	0.095	28.2

The ultrasonic velocity and attenuation for each sample were estimated using multiple back wall reverberations obtained from a normal incidence immersion inspection with a 20 MHz, 1/4” planar compression transducer. The arrival times of the back wall signals were used with the measured thickness of the samples to estimate the velocity. The time domain data was gated and a Fourier transform applied. The ratio of the back wall signal spectra was used to estimate the attenuation using the relationship:

$$\alpha(f) = -\frac{1}{2z}\ln\left(\left|\frac{\Gamma_3(f)}{\Gamma_2(f)}\right|\right)$$

where z is the sample thickness and Γ_2 and Γ_3 are the spectra of the second and third back wall, respectively. The frequency dependent attenuation results obtained for these samples can be seen in Figure 29, and the corresponding velocity results can be seen in Figure 30.

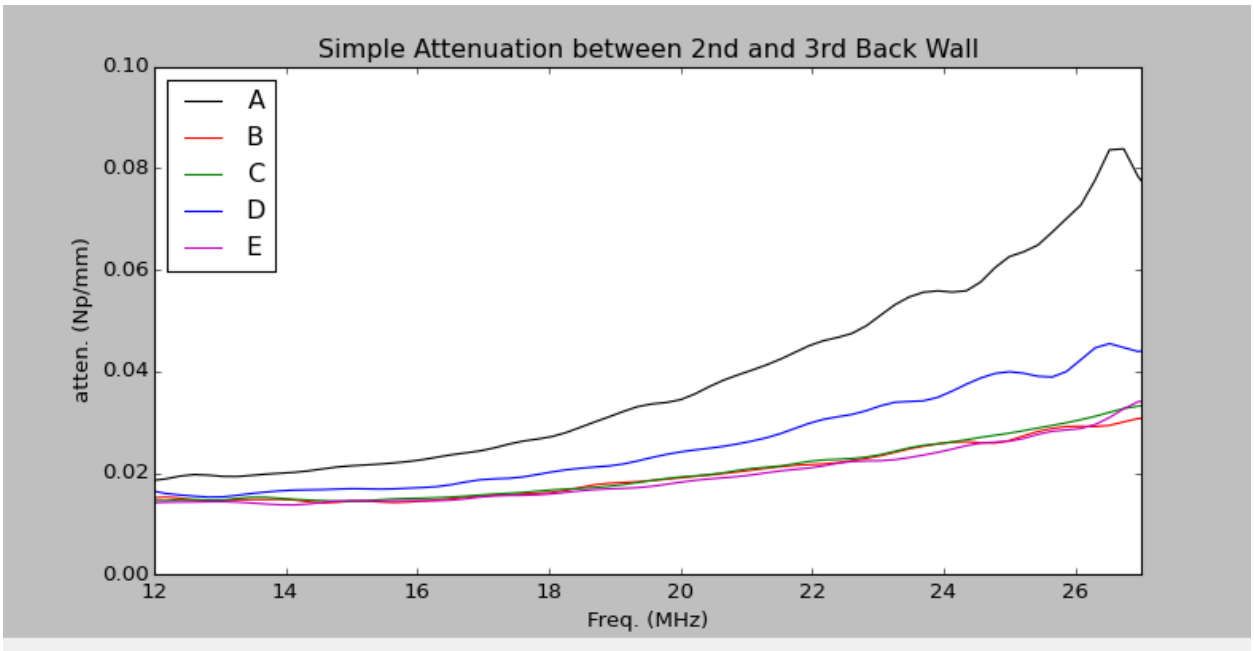


Figure 29. Attenuation curves for the controlled sample set.

There are clearly three distinct frequency dependent attenuation relationships seen in Figure 29: sample A has the highest attenuation, followed by sample D, while samples B, C, and E have similar and lower attenuation. This variability is in contrasted to the velocity results, which show that all samples have a velocity within about 0.2% of each other, which is within the estimated error for these measurements is. It was found that the attenuation of Sample F was too large to be measured at this frequency (i.e. the grain scattering was to high).

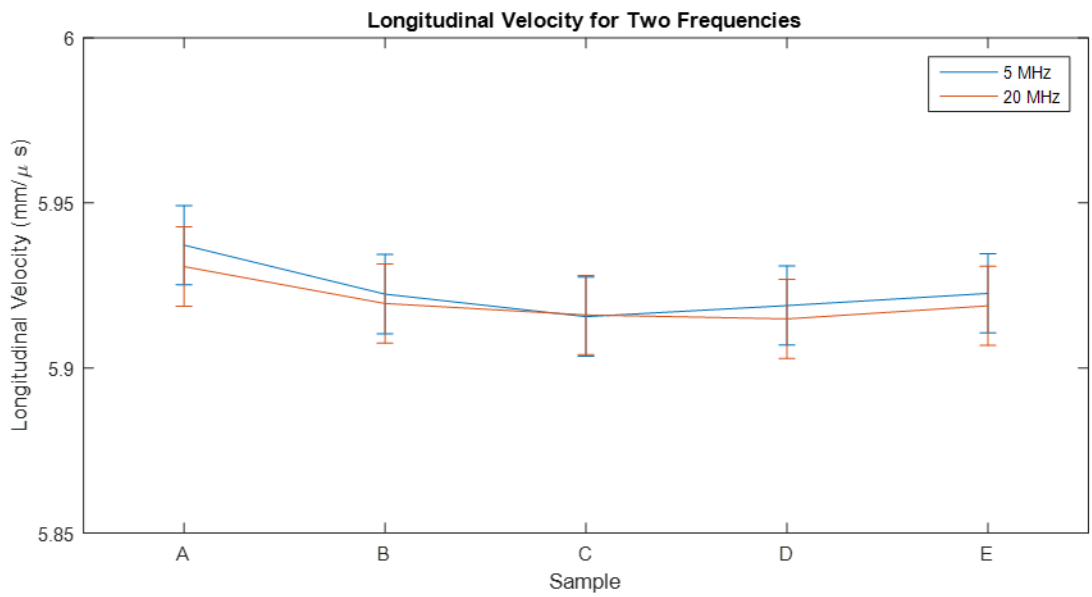


Figure 30. Velocity results for the controlled sample set.

The results of ultrasonic pulse-echo ultrasonic backscatter measurements for the various samples are shown in Figure 31. There is an anomalous feature seen for sample C that is thought to be related to the banding in the micrograph shown in Figure 32. The time domain processing in Figure 31 shows that on, on average, sample F has the largest response, followed by sample A, then D, then by B and E. This relationship is also seen in the frequency domain response which is obtained by applying a gate an an FFT to the signal. The corresponding spectra are shown in Fig 33 for the 10-20 MHz region. Sample F has the largest grains and the largest amount of measured backscatter, which was the expected result.

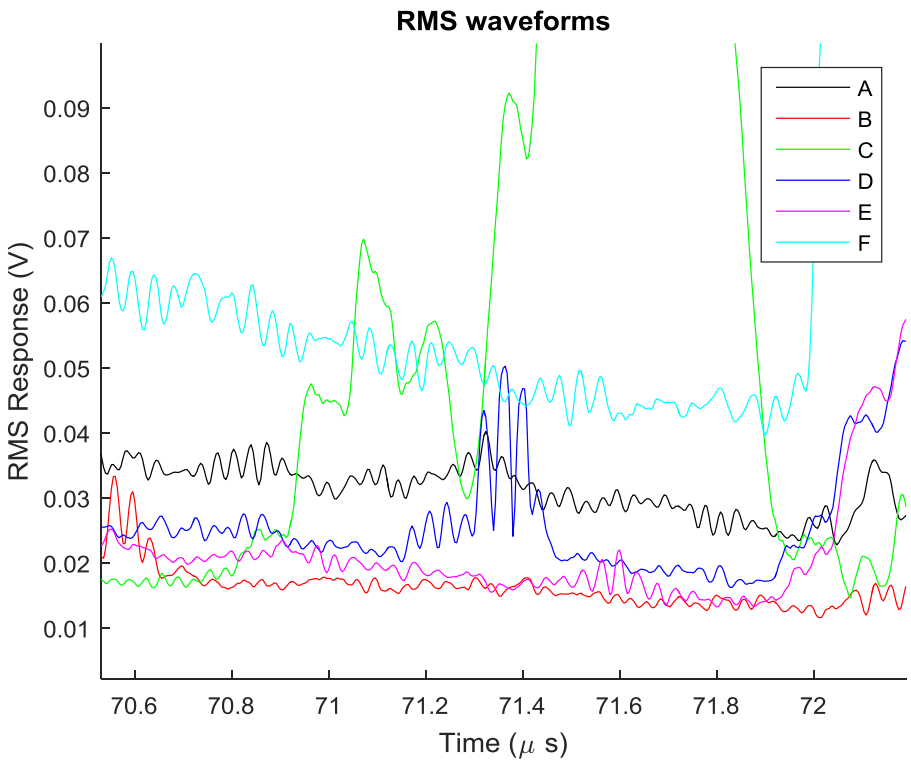


Figure 31. Backscatter results for the controlled sample set.

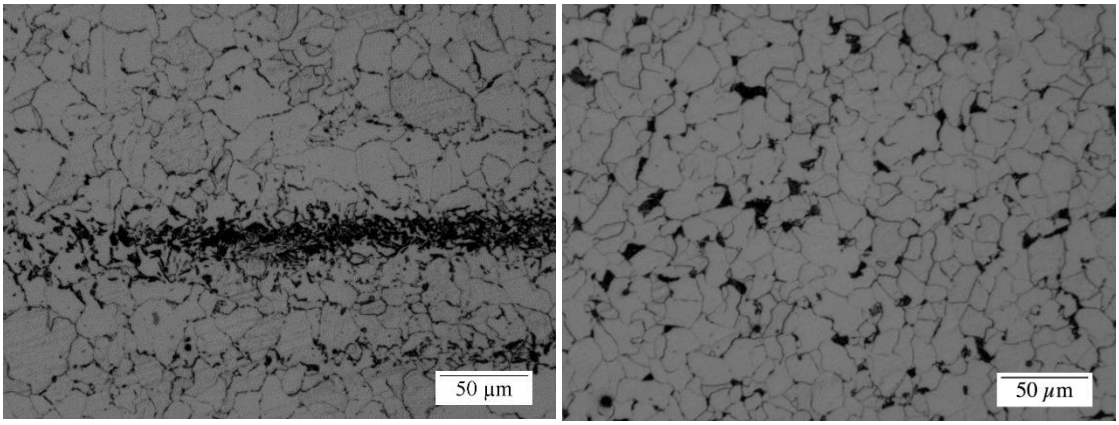


Figure 32. Micrographs for (a) sample C showing banding and (b) sample with minimal perlite. (Provided by Colorado School of Mines)

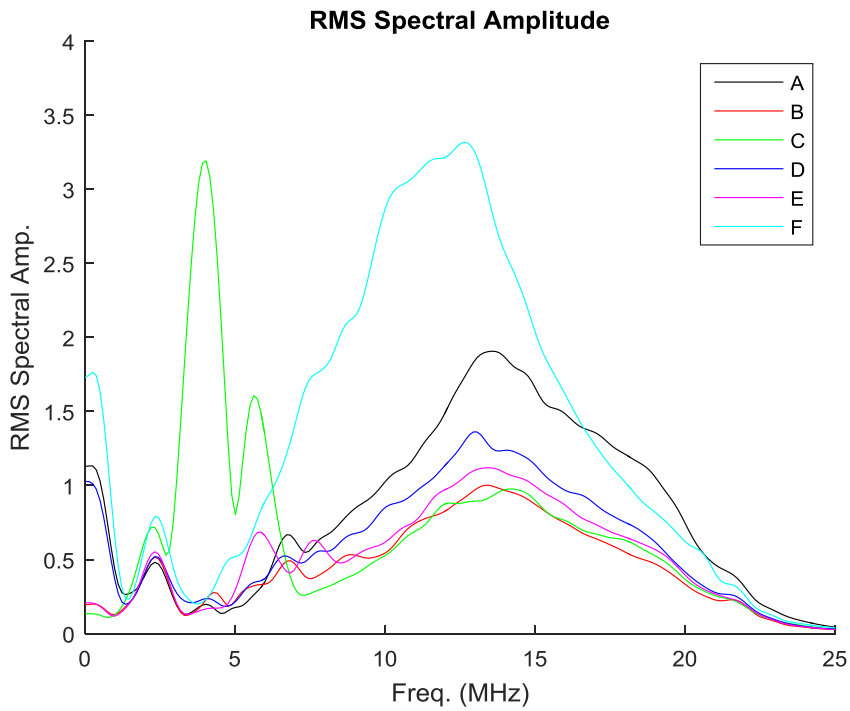


Figure 32. Frequency domain backscatter results for the controlled samples.

The Barkhausen results are shown in Figure 34 and the data show that samples A, B, and C having similar responses, while sample D has a slightly lower response, and sample E has the lowest response.

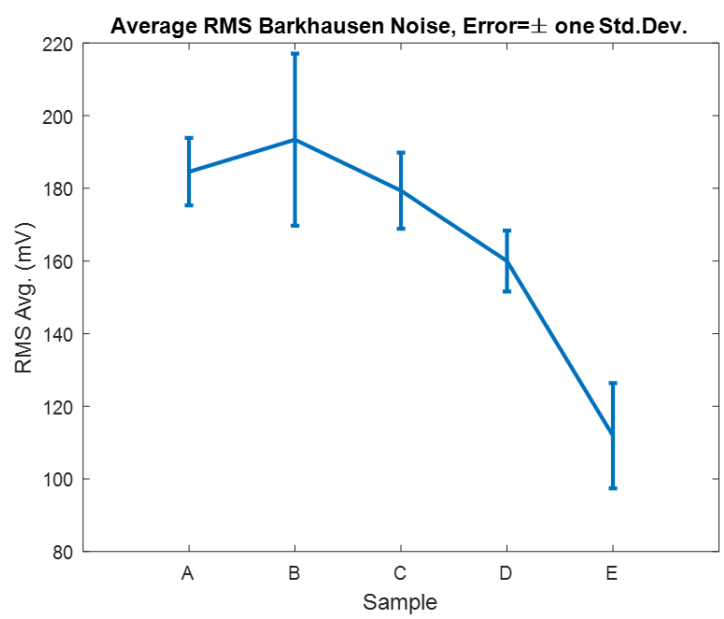


Figure 33. Barkhausen results for the controlled sample set.

It is clear from the micrograph and measured properties for the more controlled sample set that different heat treatments can have a sizable impact parameters. These samples also exhibit much more regular responses in the data for the nondestructive measurements, even on samples of the same grade, same chemical composition, and that were cut from the same base material.

7. TASK 4: CONCLUSIONS AND RECOMMENDATIONS

This project has shown that the goal of relating NDE data to mechanical properties is in reality two distinct problems that need to be decoupled in order to make further progress towards solving the over-arching problem of non-destructively mapping mechanical property determination in pipeline steels.

7.1 Material/Mechanical Property Relationships

It was shown with the data in the Kiefner data base set, which was assumed to be representative of pipeline steels in general, does not exhibit agree many of the trends common stated in the literature.

One reason could be that the samples analyzed and reported in the data base are not representative of the full range of property relationships for varying pipe vintages. It would seem that were relationships have been reported these are in general for samples of pipe from a particular mill and process. Changes and variability in processing and lack of adequate knowledge of how this related to material properties needs further study.

To make further progress there needs to be a way to relate material properties (chemistry, microstructure, etc.) to mechanical/strength properties **for the general set of pipeline steels**. This is a significant statistics and materials problem that must be solved to achieve the goal of using nondestructive measurements to estimate strength properties. Even if the nondestructive measurements gave well bounded results that allowed for complete characterization of the microstructure, this information will not give the designed goal of nondestructively determined strength measurement unless the measured microstructure can be related to the strength properties.

7.2 NDE/Material Property Relationships

An outcome of a project to determine how material properties relate to strength properties in general could be based on a modality that gives a set of key microstructural properties such as grain size and pearlite content. A further series of studies are required to examine further the effects on nondestructive results of the variation seen in properties and their relationship to strength as they relate to pipes of the same age. This would allow for clear identification of which measurement modalities are sensitive to the different microstructural parameters, for particular vintage (or specific mill sourced) pipe. This could then be further developed by examining the effects on response when two microstructural parameters are allowed to vary. The complexity could then be increased to incorporate additional metrics and more vintages and pipe

sources. With a sufficiently large and well pedigreed set of pipe material samples it would appear progress can potentially be made to unravel the multi-parameter-variability found in the general set of pipeline steels samples which were available to this project. An important consideration in this approach is understanding the manufacturing processes, its effect on pipe properties and how this has changed over time. There is also the relatedly issue of how the chemistry of pipeline steels has changed, particularly in relation to the level of carbon and manganese, which are significant in the strengthening alloys used and improved over time.

8. CONCLUSIONS

This project set out to seek to determine strength and toughness properties in pipeline steels using nondestructive measurements. Some progress was made and measurement tools identified, but issues were encountered with regard to obtaining well controlled samples and there was an unexpected disagreement with the microstructure-strength relationships previously reported in the literature, when considered in the context of a sampling of many pipe samples from different sources.

The expected relationships between material properties, chemical composition and microstructure, and mechanical properties, such as strength and toughness, were generally not seen in the available data. It is unclear if this was a problem relating to statistics and sample sources. To clarify these issues a larger set of sample data for analysis, or a set of samples focusing on one particular variable, with regards to vintage, grade, or microstructural characteristics.

The nondestructive measurements when applied to a randomly selected and sourced set of pipe samples failed to provide data that gave clear correlations between NDE measured parameters and strength properties. There was simply too much variation to draw any significant conclusions.

NDE measurements made on a small controlled sample set showed the ability of the measurements to detect changes in microstructure caused by different heat treatments when applied to the same base material. An approach of developing a well controlled samples has potential to further unravel the complex multi-variable relationships between pipe strength parameters and NDE measurements that characterize microstructure and related electrical and mechanical properties

To make further progress it must first be possible to understand the multi-parameter effects on the large variation seen in in-service pipe samples. There needs to be a way estimate the strength properties if microstructure, chemistry, and hardness are known for the general set of pipeline steels. The nondestructive measurements need to start with more controlled sample sets in order to isolate and separate the effects of different variables and to study the effects of each one on measured data. No one NDE tool is going to give an adequate correlation between measured NDE data and strength parameters. It does appear that with several electrical and ultrasonic measurements progress could be made.

PROJECT PUBLICATIONS AND PRESENTATIONS

Engle, B.J., Smart, L.J. and Bond L.J. ((2015) Nondestructive characterization of pipe materials. Proceedings 41st Annual Review of Progress in QNDE, Ed. D.E. Chimenti and L.J. Bond, AIP (New York) AIP Proceedings # 1650, pp 943-951

Bond, L.J. (2015) Needs and opportunities: nondestructive evaluation for energy systems (Keynote Presentation), Proc. SPIE 9439, Smart Materials and Nondestructive Evaluation for Energy Systems 2015, 943902 (March 27, 2015); doi:10.1117/12.2086279

Engle, B.J., Smart, L.J., Bond L.J. (2016) Ultrasonic and magnetic Barkhausen emission measurements for characterizing pipeline steels, Nondestructive characterization of pipe materials. Proceedings 42st Annual Review of Progress in QNDE, Ed. D.E. Chimenti and L.J. Bond, AIP (New York) AIP Proceedings [In Press].

Smart, L & Bond, L.J. (2016) Material Property Relationships for Pipeline Steels and the Potential for Application of NDE, Proceedings 42st Annual Review of Progress in QNDE, Ed. D.E. Chimenti and L.J. Bond, AIP (New York) AIP Proceedings [In Press].

Smart, L (2015) Literature study for nondestructive characterization of pipeline materials. MS Thesis, Mechanical Engineering Department, Iowa State University.

REFERENCES

Belanger, A., and Barker, T. "Multiple Data Inspection of Hard Spots and Cracking." 2014 10th International Pipeline Conference. IPC2014-33060. Calgary, AB, Canada: ASME, 2014.

Cooreman, S., Hoecke, D.V., Liebeherr, M., Thibaux, P. and M.Y. Enderlin. M.Y. "Measurement of Mechanical Properties on Line Pipe: Comparison of Different Methodologies." 2014 10th International Pipeline Conference. IPC2014-33520. Calgary, AB, Canada: ASME, 2014.

Ensminger, D. and Bond, L.J. “Ultrasonics: fundamentals, technology, applications, 3rd Revised Edition”, CRC Press, Taylor and Francis Group (Boca Raton) 2011.

Gray, J. M., and Siciliano F.. "High strength microalloyed linepipe: half a century of evolution." Microalloyed Steel In stitute (2009): 20-45.

Jiles, D. C. "Review of magnetic methods for nondestructive evaluation." NDT international 21.5 (1988): 311-319.

Kiefner, J.F. and Clark, E.B. History of Line Pipe Manufacturing in North America. Gas Pipeline Safety Research Committee, ASME, 1996.

Kiefner, J.F. and Rosenfeld, M.J. The Role of Pipeline Age in Pipeline Safety. INGAA Foundation, Inc, 2012.

Krauss, G.. Steels : Processing, Structure, and Performance. ASM International, 2005.

Margetan, F. Barnard, D., Orman, D., Feygin, A. and Pavel, B. “A Web-Based Tutorial for Ultrasonic Attenuation Measurement,” Proceedings 40th Annual Review of Progress in Quantitative Nondestructive Evaluation, AIP Conf. Proc. 1581, 2127-2134, 2014

Molenda, D. and Thale, W.. "A Novel Approach for Pipe Grade Determination Through In-Line Inspection (ILI)." 2014 10th International Pipeline Conference. IPC2014-33441. Calgary, AB, Canada: ASME, 2014.

National Transportation Safety Board. 2011. *Pacific Gas and Electric Company Natural Gas Transmission Pipeline Rupture and Fire, San Bruno, California, September 9, 2010*. Pipeline Accident Report NTSB/PAR-11/01. Washington, DC.

Raj, B., Moorthy, V., Jayakumar, T. and Bhanu Sankara Rao, K.. “Assessment of Microstructures and Mechanical Behaviour of Metallic Materials through Non-Destructive Characterisation.” International Materials Reviews 48, no. 5 (2003): 273–325.

Samuels, L. E.. Light Microscopy of Carbon Steels. ASM International, 1999.

Thompson, R. B. "Ultrasonic measurement of mechanical properties."Ultrasonics Symposium, 1996. Proceedings., 1996 IEEE. Vol. 1. IEEE, 1996.

Thompson, R. B.. "Quantitative ultrasonic nondestructive evaluation methods." Journal of Applied Mechanics 50.4b (1983): 1191-1201.

Zhao, M.C., Yang,K. and Shan. Y."The effects of thermo-mechanical control process on microstructures and mechanical properties of a commercial pipeline steel." Materials Science and Engineering A335, 2002: 14-20.