

BI-MONTHLY PROGRESS REPORT - III

**NEW METHODOLOGIES FOR MEASURING AND MONITORING HYDROGEN
FOR SAFETY IN ADVANCED HIGH STRENGTH LINE PIPE STEEL**

**Development of a Non-Destructive, Non-Contact Electromagnetic Sensor
for Hydrogen Determination in Coated Line Pipe Steel**

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1. Introduction

Productivity, safety, and economics are driving a new paradigm of pipeline materials and construction by trying to achieve pipelines with higher strengths that are operating at higher pressures and with larger diameters. The demand on the increase in strength makes the pipelines vulnerable hydrogen-assisted cracking. Mitigation of hydrogen damage is essential for pipeline integrity, thus making advanced development of non-contact, non-destructive hydrogen sensing technology essential.

This bi-monthly report will outline the necessary steps for calibration and measurement of three non-destructive, non-contact, electromagnetic and tools for determination of hydrogen content in coated line pipe steels and characterize the specific temperature and steel that is being measured. The necessity of the utilization of each electromagnetic and acoustic tool for an accurate determination of hydrogen content will also be explained.

2. Development of Non-Contact Hydrogen and Microstructural Sensors

This report period has been devoted to the development of a measurement practice to accurately monitor and measure the diffusible hydrogen content in coated line pipe steels using a non-destructive, non-contact tool. Electronic property measurements have demonstrated their ability to assess diffusible hydrogen content in steel with a prior calibration of the electronic signal relative to a known hydrogen standard of that steel and the temperature of the pipe. Since electronic properties have a number of dependent physical and compositional variables, it is essential to utilize other non-contact measurements to characterize the steel and its temperature. Hydrogen standards will be made for specific combinations of equivalent steel microstructures and temperatures for a variety of line pipe steels. The electronic property measurement will be correlated to the hydrogen content for these hydrogen charged steel standards.

This investigation is developing a practice of using a combination of three separate measurements to allow the hydrogen content to be rapidly determined for a specific equivalent steel microstructure and pipeline temperature. Eddy current analysis will be the primary method to assess the hydrogen content. The speed of sound in the pipe will be measured to assess the pipe temperature using ultrasonic wave induction and receiving using EMAT technology to determine the time of travel between a known distance. From this, the velocity of sound in the steel can be determined and correlated to temperature of the pipe. Magnetic Barkhausen Noise (MBN) will be used to assess for equivalent steel microstructures. The MBN technique causes magnetic domain walls to sweep through the steel where the traveling block wall interacts with the steel microstructural features such as: grain boundaries, precipitates, etc. The magnetic cyclic perturbation will be induced non-destructively and with non-

contacting eddy current coil. The interaction of the domain wall with microstructural features will be measured as a function of induced frequency. The elastic wave emissions as the domain wall pulls away from microstructural pinning sites will be measured as a function of frequency. These emissions will be assessed with the use of non-contact EMAT acoustic receiver. The signals of specific microstructural pinning features will be exhibited at certain applied eddy current frequencies. Thus, the microstructural features in the steel can be characterized and identified as a specific equivalent microstructure. The more complete description of the practice using these three non-contact, non-destructive hydrogen analytical tools is discussed below.

2.1. Eddy Current Hydrogen Analysis

To meet the requirements of a non-destructive, non-contact measurement practice, eddy current analysis will be used for assessment of hydrogen content in coated line pipe steel. Klümpfer-Westkamp et al. [2003] has shown that eddy currents with a harmonic analysis can be used to assess carburization depth in iron foils (Figure 2). An eddy current with a harmonic analysis is a non-destructive method and has been adapted in a small automated device, which is robust and simple to use. With this new device the carbon content of thin iron foils can be obtained non-destructively with accuracy of better than ± 0.03 wt. pct. carbon in less than two seconds within the range of 0 - 1.2 wt. pct. carbon content [Klumper-Westkamp, 2002]. These results are very valuable in giving insight and encouragement for the design of a measurement scheme with the sensitivity to measure diffusible hydrogen.

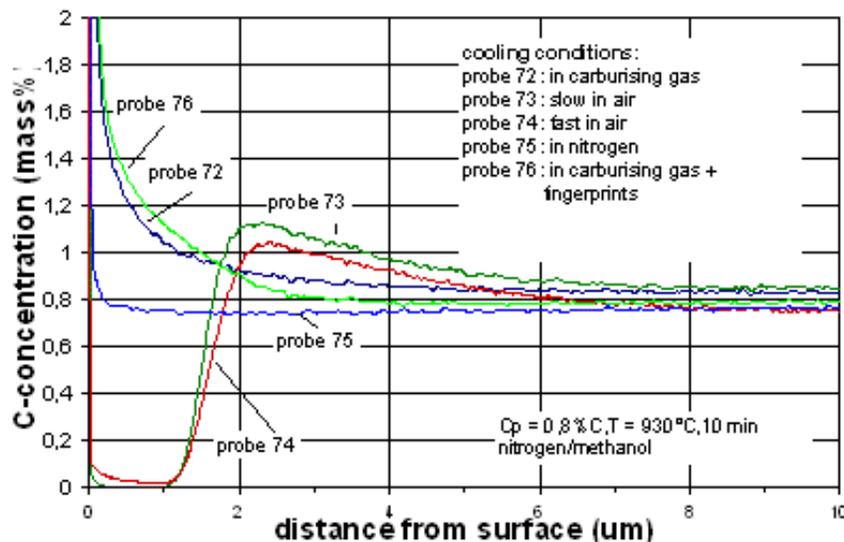


Figure 2: Eddy current analysis of carbon concentration as a function of distance from surface for measuring case depth [Klumper-Westkamp et al., 2003].

Griffith et al [1997] utilized an eddy current unit encircling an insulated pipeline for detection of cracks and defects in oil and gas pipelines as shown in Figure 3. These same encircling coils can potentially be used to assess hydrogen content in coated steel pipelines.

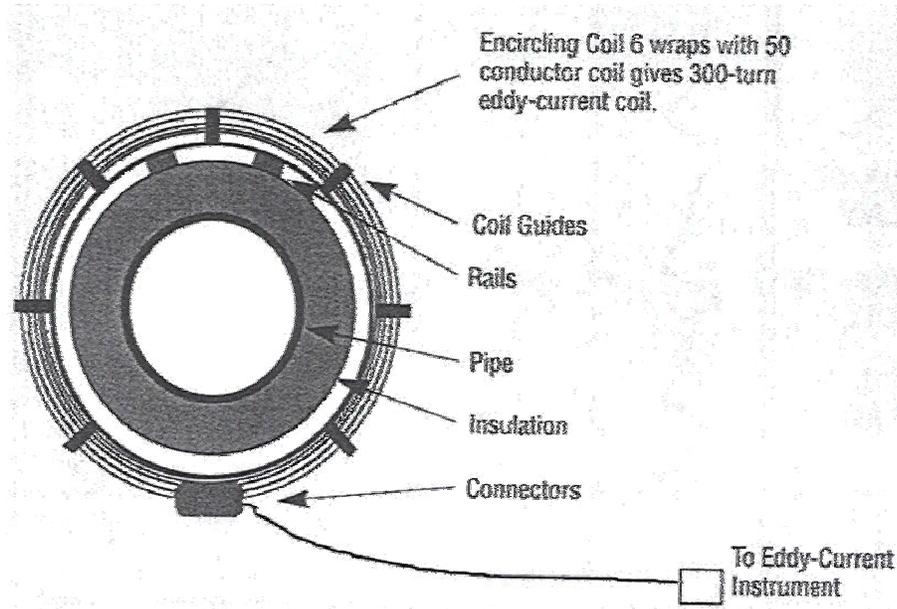


Figure 3: Eddy current unit encircling an insulated pipeline [Griffith et al 1997].

2.1.1. Complex Functionality of Eddy Currents

Reflecting back on the past reports, the hydrogen content can be assessed by correlating it to the measured phase shift between the reactants and the resistance as shown in Equation 1.

$$\alpha = \tan^{-1} (\omega L - 1/\omega C)/R \quad [1]$$

where L is inductance, C is capacitance, ω is angular velocity, and R is resistance. However, the phase shift is a very complex variable, being a function of conductivity, which directly influenced by the electron concentration, the effective mass, and scattering sites. The phase shift is also a function of temperature and alloy content.

According to Matthiessen's rule [Kittel, 1996] the resistivities from various electronic structure interactions in a material are additive, so that the total resistivity in a material is given as:

$$\rho = \rho_T + \rho_D + \rho_{GB} + \rho_H \quad [2]$$

where ρ_T is resistivity due to temperature, ρ_D is resistivity due to defects, ρ_{GB} is resistivity due to grain boundaries, and ρ_H is resistivity due to hydrogen. To measure the change in resistivity due to only hydrogen, it is necessary to account for the effect of temperature, defects, and grain boundaries through the use of other electromagnetic and acoustic techniques.

The resistivity due to temperature can be accounted for by utilizing EMAT measurements and the resistivity due to defects and grain boundaries can be accounted for by utilizing Magnetic Barkhausen Noise measurements as described below. Thus for a steel with a specific equivalent microstructure and temperature, the hydrogen content can be correlated to the phase shift α with the known data from hydrogen standards of an equivalent microstructure and temperature. Such a correlation is suggested by the shift in phase angles with hydrogen content as shown in Figure 4.

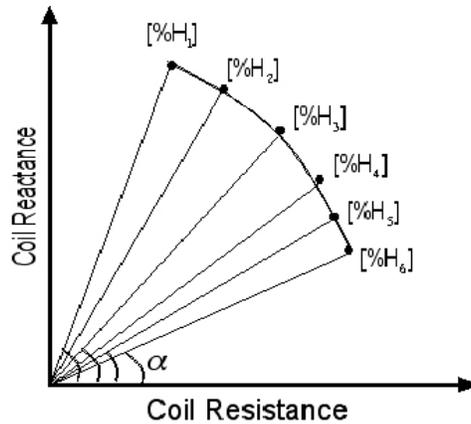


Figure 4: Schematic diagram of coil reactance as a function of coil resistance showing the change in phase shift, α , with variations in hydrogen concentration in line pipe steel.

2.2. EMAT Analysis to Account for Temperature Effect

Pipelines around the world experience various environmental conditions, which must be accounted for to achieve an accurate hydrogen measurement with eddy current analysis. Variations in pipeline temperatures will give an inaccurate assessment of hydrogen content, thus making it necessary to have a non-contact measurement practice that accounts for the effect of temperature as discussed above in Equation 2.

Two electromagnetic acoustic transducers (EMAT's) will be placed at a fixed distance apart where one EMAT will transmit an ultrasonic wave pulse, while the

other EMAT receives the ultrasonic wave pulse as shown in Figure 5. The speed that it takes for the ultrasonic wave pulse to travel this fixed distance is a function of the material, which is temperature dependent, thus allowing for a method to remove temperature as a variable in the determination of the hydrogen content. Resistivity due to temperature has been accounted for by using EMAT speed of sound analysis. The defects, grain boundaries, and microstructural features unique to every pipeline must also be accounted for to achieve an accurate hydrogen measurement.

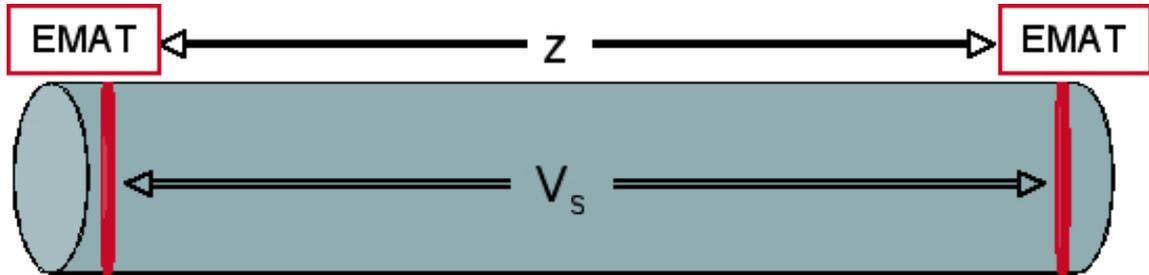


Figure 5: Schematic illustration of set-up of electromagnetic acoustic transducers (EMAT's), for speed of sound, V_s , measurements at a distance Z apart.

2.3. Magnetic Barkhausen Noise Microstructural Analysis

Magnetic Barkhausen Noise has been highlighted for evaluation of microstructural features in ferromagnetic materials. Ferromagnetic materials consist of magnetic domains in which the material is magnetically saturated. The domains are separated from one another by boundaries, which are called domain walls. The elementary magnetic moments change continuously across these domain walls from spin up to spin down [Gintsztler et al, 1994].

If the strength and direction of the external magnetic field changes, the domain walls will move. The volume of the domains in which the magnetization is nearly parallel to the external magnetic field increases at the expense of other domains. The domain wall movement (resulting from eddy current induction) is hindered by microstructural features for which the domain wall experiences pinning and their passage will produce an elastic pulse (Barkhausen Noise), which can be measured with an acoustical non-contact sensor (EMAT). The elastic pulse for a specific feature is a function of the induced frequency from the eddy current coil. Performing a frequency analysis on these Magnetic Barkhausen signals will allow for characterization of the given pipeline steel as a specific equivalent steel microstructure. The correlation of a set of hydrogen charged specimens to a specific equivalent steel microstructure will serve as a standard in the determination of the diffusible hydrogen content in coated steel line pipe.

For Magnetic Barkhausen Noise analysis, the eddy current coil is used to induce the magnetic domain movement and the EMAT can be utilized to listen to the acoustoelastic signal as shown in Figure 6.

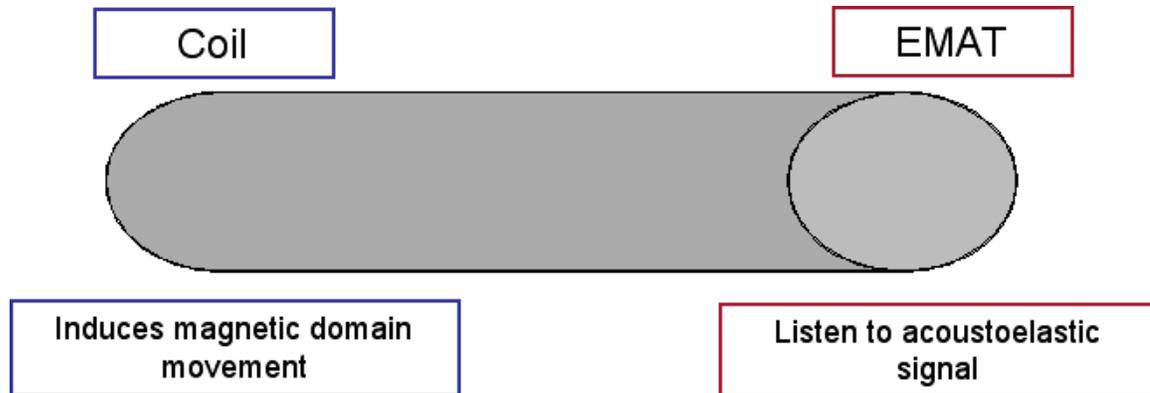


Figure 6: Schematic illustration of Magnetic Barkhausen unit utilizing the eddy current coil and the electromagnetic acoustic transducer.

3. Current Work and Tasks

The advantage of the electromagnetic instrumentation described above is that it is completely a non-contact, non-destructive measurement system. CSM is collaborating with CANDET in Canada for the development of an electromagnetic non-contact hydrogen sensor containing an eddy current unit for hydrogen measurements, EMAT analysis to account for temperature, and MBN to account for microstructure. This collaboration requires transport of hydrogen-charged specimens to CANDET for NDE analysis. The transport of hydrogen-charged specimens requires a proper coating/plating technique to serve as a hydrogen barrier, thus maintaining the hydrogen content in the specimens. CANDET will use these specimens to guarantee electromagnetic equipment sensitivity. Once the necessary sensitivity is achieved, CANDET will build and send an electromagnetic box unit to CSM for calibration of specimens for hydrogen content measurements. Notice that the instrumentation used in these electromagnetic techniques can be used together, which is important for economics and efficiency. For example, in Magnetic Barkhausen Noise analysis the eddy current coil is used to induce the magnetic moment and the electromagnetic acoustic transducer receives the signal.

The following section describes the order of tasks necessary for the development and calibration of a non-contact, non-destructive electromagnetic and acoustic measurement system.

3.1. Hydrogen Charging and Plating

To hydrogen charge specimens, a high-pressure gaseous hydrogen and high temperature charging system has been built. Various combinations of temperature, pressure, and cooling rates can be achieved with this charging system. Once a sample has been hydrogen charged, the hydrogen content is measured using a LECO RH-404 Hydrogen Determinator. Currently, API-X80 grade line pipe steel specimens are being charged to various levels of hydrogen content to develop a hydrogen-charging scheme.

For electromagnetic analysis, specimens are hydrogen charged and then, to maintain the hydrogen content in the sample, the specimens are plated/coated. Three different platings are being experimentally tested for change in hydrogen content as a function of time. These coatings/platings include: electroless copper plating, electroless nickel plating, and a non-cyanide cadmium electroplating. The plating that best maintains the initial hydrogen charging content for the longest period of time will be utilized for transportation of samples to Canada. This work is in progress. If the hydrogen charged specimens cannot be transported without maintaining constant hydrogen content by July 1, 2005, then CSM will send an investigator to hydrogen charge the specimens at the CANDET Research Lab in Toronto, Canada.

The measurement scheme for hydrogen determination has been described and now it is important to discuss the tasks for calibration and measurement of hydrogen content in coated line pipe steel.

3.2. Tasks for Electromagnetic and Acoustic Hydrogen Analysis

To guarantee an accurate measurement of diffusible hydrogen content in steel it is important that the eddy current hydrogen analysis system be properly calibrated for each particular steel sample under specific conditions. The steps to calibration are also notable and must be considered very carefully to achieve the most sensitive hydrogen measurement. This report period has been spent in designing a calibration and charging system.

Before an electromagnetic and acoustic measuring scheme can be designed, the calibration of each individual electromagnetic and acoustic technique is necessary. Eddy current analysis must be calibrated for measurement of hydrogen, EMAT analysis must be calibrated for speed of sound measurements, and MBN analysis must be calibrated for microstructure (scattering sites). After the calibration steps are described, the measurement scheme to complete a thorough analysis to achieve an accurate hydrogen measurement through line pipe coating is discussed.

3.2.1. Eddy Current Analysis Calibration

Eddy current analysis for hydrogen assessment must be calibrated for each specific type of steel. Every alloy will have its own signature change in phase shift with varying hydrogen contents. To calibrate the eddy current analysis for use on X80 steel specimens, the specimens are charged to various levels of hydrogen contents, then coated and allowed to homogenize for 48 hours. Eddy currents measurements will be made on each X80 steel specimen charged with a different hydrogen content to determine the associated phase shift as schematically shown in Figure 4. Phase shift results for numerous X-class alloys of various thermal experiences are stored into the database.

3.2.2. EMAT Analysis Calibration

The electromagnetic acoustic transducer speed of sound analysis must be properly calibrated for an X80 steel specimen. The speed of sound will be measured on the X80 steel specimen as a function of temperature. Every steel alloy will have its own signature speed of sound as a function of temperature and must be properly calibrated for the alloy of interest. Eddy current calibration should be performed on numerous X-class alloys and thermal experiences to build a database for speed of sound as a function of temperature. It is important that numerous X-class alloys and thermal experiences be calibrated because different pipelines will be made from different alloys in different environments, thus making it necessary to have a calibration measurement for each alloy before a hydrogen assessment can be made.

3.2.3. Magnetic Barkhausen Noise Analysis Calibration

Magnetic Barkhausen Noise analysis must also be properly calibrated for the X80 steel specimen. X80 steel specimens will be calibrated with Magnetic Barkhausen Noise by scanning the specimen at a range of frequencies to determine the frequency array associated with the X80 specimen. Every steel alloy will have its own signature frequency range because each alloy has its own distinct scattering sites. Magnetic Barkhausen Noise calibration measurements will be performed on numerous X-class steel alloys and thermal experiences to characterize the different steels with their own microstructural (scattering) signal as discussed above. The results for the calibrated X-class steel alloys and thermal experiences are put into the computer database.

A number of X-class steel alloys with different compositions and thermal experiences will be used to demonstrate the above practice of using three techniques to assess hydrogen content for equivalent steel microstructure and temperature as shown in Figure 7.

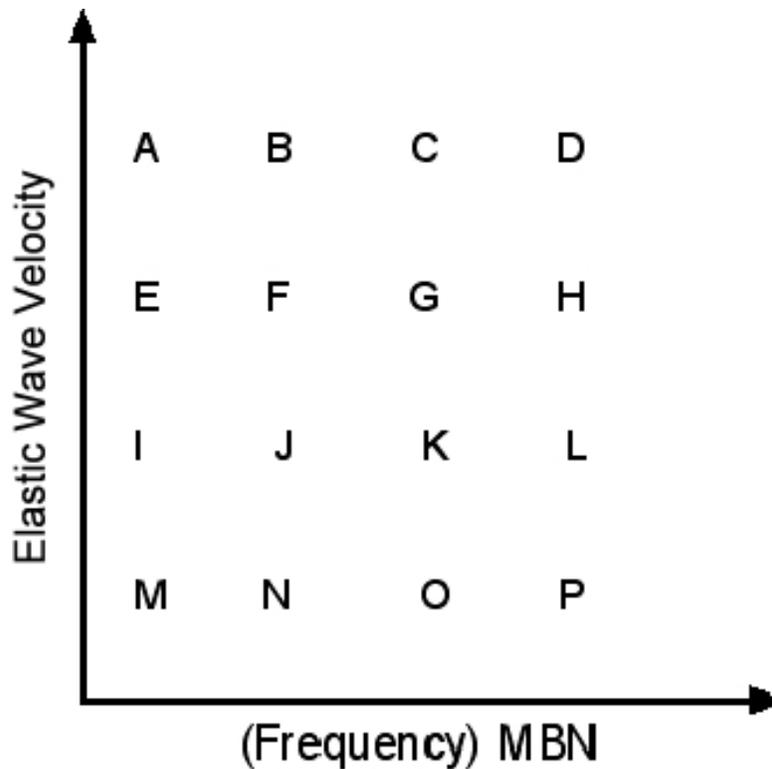


Figure 7: Schematic diagram of line pipe characterization through the elastic wave velocity (from EMAT) as a function of the product of frequency (from MBN) and Magnetic Barkhausen noise.

3.3. Measurement Scheme for Electromagnetic Analysis of Hydrogen in Coated Line Pipe Steel

Assuming that calibration of all three electromagnetic techniques has been performed (described above), Table 1 lists the steps necessary to obtain an electromagnetic assessment of hydrogen content in line pipe X80 steel specimens at the Colorado School of Mines. There are two different X80 steel specimen sizes being utilized in this particular experimental analysis for convenience. (Note: During charging the hydrogen reactor will contain both a small and a large cylindrical specimen) Small cylindrical samples with a length of 1 cm and a diameter of 0.3 cm are used for the Leco hydrogen determinator to measure the hydrogen content in the X80 steel specimen. The larger cylindrical specimens are 4 cm in length and 0.5 cm in diameter and will be used for the electromagnetic analysis. Different specimen sizes are necessary to quickly analyze the X80 steel specimens for both hydrogen content in the Leco Hydrogen Determinator and using electromagnetic analysis.

Table 1: Steps for electromagnetic hydrogen analysis.	
Task #	Task
1	Hydrogen charge X80 steel specimens at a specific pressure and temperature to achieve a certain hydrogen content
2	Remove specimens from hydrogen charging reactor and quench in liquid nitrogen
3	Small cylindrical specimen is immediately put into the Leco Hydrogen Determinator for hydrogen content determination
4	Large cylindrical specimen is quickly electroplated to the maintain hydrogen content (determined from Task 3) in the X80 steel specimen
5	Large cylindrical X80 specimens are annealed at 200°C for 48 hours to homogenize the hydrogen content in the X80 steel specimen
6	Eddy current analysis is performed to determine hydrogen content in the line pipe X80 steel specimen
7	EMAT speed of sound analysis is performed to account for temperature
8	MBN analysis is performed to account for microstructure (scattering)
9	Data received from Steps 7-8 is compared with the database of calibrations and the steel is associated with a specific equivalent steel class type (Class A, B,C). Schematically shown in Figure 7.
10	Then the eddy current analysis calibration database for this specific equivalent steel class type (e.g. Class A) is used to determine the final hydrogen content. Schematically shown in Figure 8.

Figure 8 illustrates how the measured eddy current signal will be compared to measured hydrogen standard for an equivalent steel microstructure and temperature to achieve hydrogen content in the coated line pipe steel specimen.

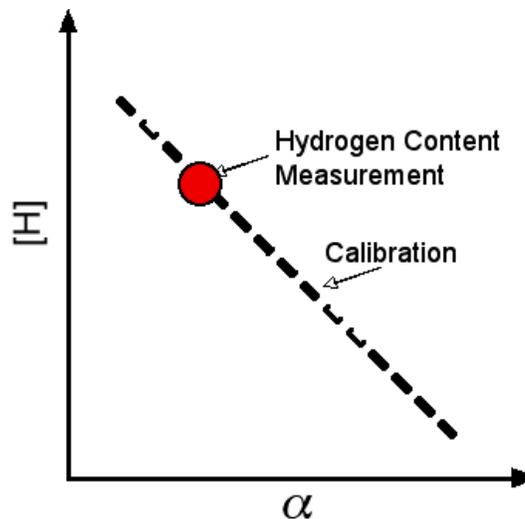


Figure 8: For a given steel class from Figure 9, the hydrogen content is determined from a plot of hydrogen content as a function of the phase shift.

4. Thermoelectric Power Assessment of Line Pipe Steel Weld Metal Microstructural Evolution and Diffusible Hydrogen Assessment

The thermoelectric power research task, for assessing weld hydrogen content as described in the second bi-monthly report, has changed its focus. This change in focus is due to the requirement that hydrogen needs to be non-destructively assessed through the pipeline coating. Thermoelectric power uses a non-destructive surface contact probe, meaning that it cannot sense hydrogen through the coating. The effort to assess hydrogen through the coating on the steel pipe is now to be addressed with the eddy current analytical approach.

Early work has demonstrated that thermoelectric power can assess both the diffusible hydrogen content in steel as well as the time-temperature microstructural evolution behavior of the weld deposit following weld solidification. This microstructural evolution assessment can be very valuable to give quality assurance for weld repair of the pipeline during field welding. A Masters of Science graduate student has been added to this task to demonstrate the value of using thermoelectric power analysis to increase the integrity of field weld repair.

An example of recent thermoelectric power research for assessment of hydrogen content is discussed below.

4.1. Hydrogen Content During Heat-Up of Cryogenic Steel Weld Metal Specimens

The thermoelectric power coefficient was measured on HSLA steel welds as a function of specimen temperature for various diffusible hydrogen contents in the HSLA steel welds as shown in Figure 9. Each curve shows an approximately linear relationship between the TEP coefficient and the sample temperature when the TEP coefficient is plotted as a function of sample temperature. This data indicates that each TEP coefficient curve represents an amount of diffusible hydrogen content in HSLA steel welds. Therefore, by recording both the TEP coefficient value and specimen temperature for a diffusible hydrogen content measurement practice, instantaneous amounts of diffusible hydrogen for weld metal can be obtained. The TEP coefficients at a fixed sample temperature such as $-40\text{ }^{\circ}\text{C}$ are taken and are plotted as a function of diffusible hydrogen content. The results are shown in Figure 10. The results indicate that TEP coefficient is relatively linear with amount of diffusible hydrogen in welded HSLA steel at a sample temperature of $-40\text{ }^{\circ}\text{C}$ [Park, 2003].

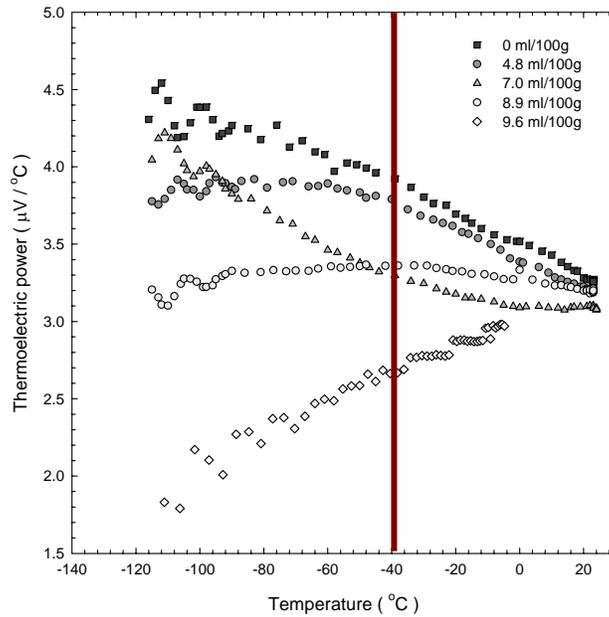


Figure 9: Measured TEP coefficient as function of specimen temperature for all five welded HSLA steel specimens [Park, 2003].

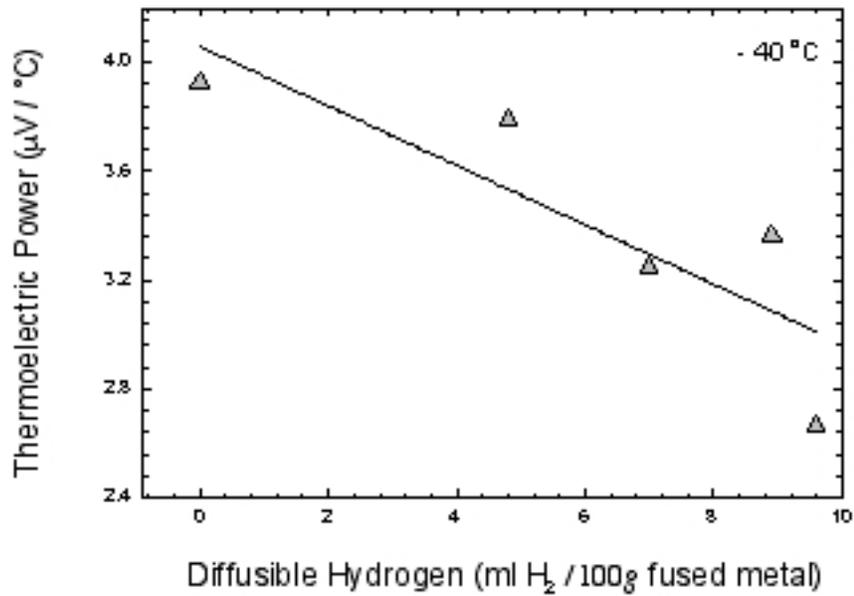


Figure 10: Measured TEP coefficient as a function of hydrogen content vol. pct. in shielding gas at specimen temperature of $-40\text{ }^{\circ}\text{C}$ [Park, 2003].

4.2. Steel Weld Metal Phase Transformations on Cooling

With a reduction in temperature with cooling the real lattice will contract and the reciprocal lattice space will expand resulting in two changes for the electronic state for transition metal alloys. The first change is the amount of d-orbital overlap between lattice atoms resulting in changes in the electron concentration, and thus the Fermi energy, in the d-band as well as a change in the shape of the d-band. The second change in the electron concentration results from the contracting real lattice, which also increases the Fermi energy level.

The TEP surface contact probe of as-deposited weld can be used to characterize the weld metal phase transition as a function of temperature and time on cooling. The arrangement illustrated in Figure 11 can be used to make this assessment. Figure 12 illustrates the resulting signatures of phase changes on weld metal cooling. This result is part of a preliminary investigation. The signature, which characterizes the evolution of weld microstructure, and thus properties, can be further enhanced by use of a differential circuit function during data acquisition. These signatures can be used in quality assurance of specific process parameters to guarantee that the required properties are being achieved.

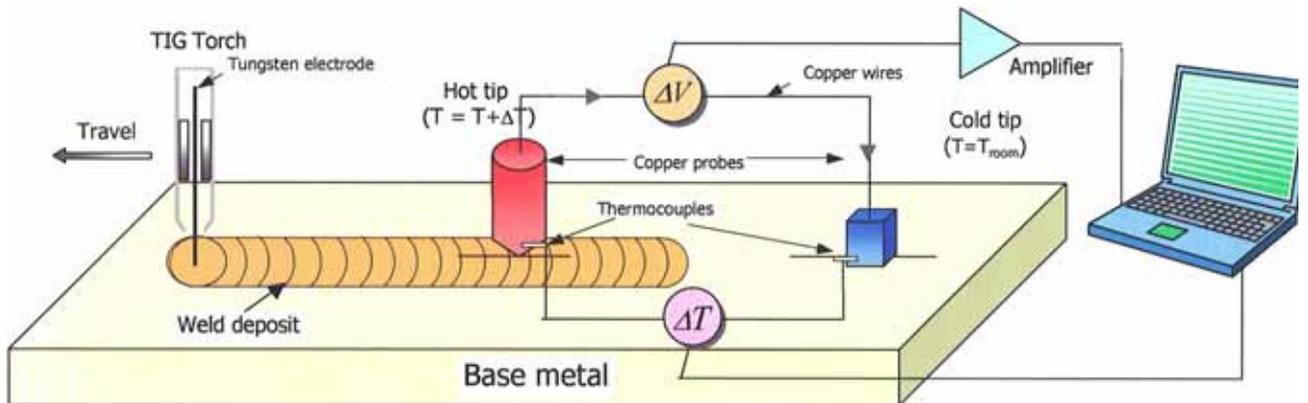


Figure 11: Schematic illustration of thermoelectric power set-up for performing thermoelectric power cooling measurements with the hot probe located in the center of the weld bead [Park, 2003].

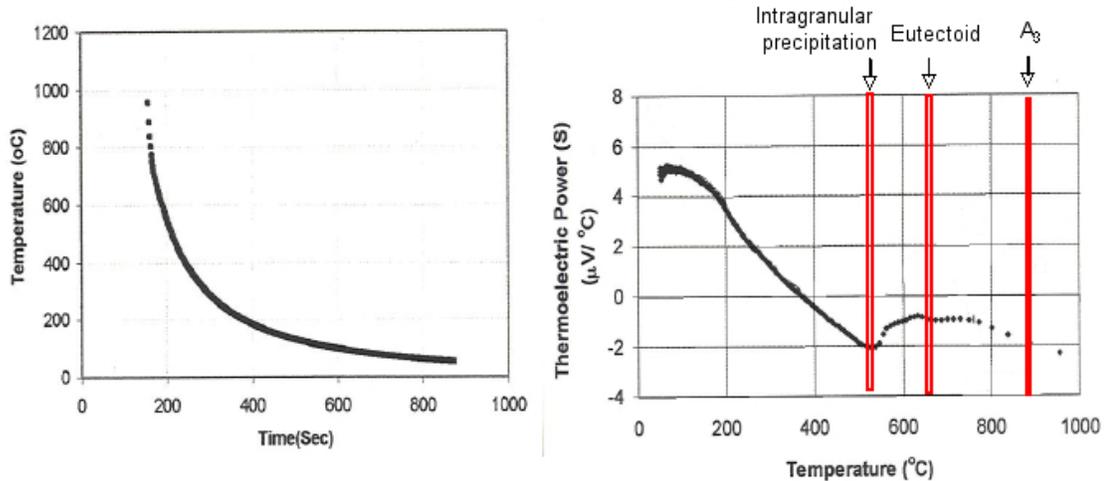


Figure 12: Thermoelectric power measurements on HSLA steel weld metal (a) temperature as a function of time and (b) thermoelectric power coefficient as a function of temperature [Park, 2003].

5. Accomplishments During the Bi-Monthly Progress Period of March 1 - April 30, 2005

1. The practice to hydrogen charge X80 line pipe steel specimens to specific hydrogen contents is being perfected. Also, the practice of poisoning (hydrogen diffusion barrier) these specimens, so that they can be transported to CANDET in Toronto, Canada. CANDET will be perfecting the eddy current analysis with these specimens. After successful measuring of hydrogen in line pipe with eddy current, the eddy current analytical equipment will be delivered to CSM. The practice to charge has been perfected, but efforts are still continuing on the electroplating of the charged specimens to make a diffusion barrier.
2. CANDET firm is working on the design for the three instrument assessment of coated line pipe steel to measure diffusible hydrogen content and characterize the steel.
3. The thermoelectric power task to measure hydrogen in line pipe steel has demonstrated that thermoelectric power can measure diffusible hydrogen with the proper analytical practice. This task has been modified to look at weld repaired X-class line pipe steel to assess both hydrogen and the microstructural evolution of the weld deposit for quality assurance. This modification has been made since thermoelectric power analysis is a surface contact analysis and the line pipe hydrogen sensor is to be used on coated line pipe steel. Since the weld repair allows exposed metal for a surface contact measurement, the achievements that we have accomplished with thermoelectric power can be furthered for weld repair

- of line pipe steel. Another research assistant, which is not at cost to MMS or DOT, will be performing the thermoelectric power analysis of weld repair deposits.
4. A paper on the fundamentals of thermoelectric power assessment for microstructure and hydrogen was prepared and will be presented at the ASM Trends in Welding Research 2005 Conference at Pine Mountain, Georgia, May 16-20, 2005.
 5. More details on these accomplishments can be found in this bi-monthly report and the attached paper.

6. References

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