Application Note

# Analysis of microalloying elements in steel using the Thermo Scientific Niton XL5

Brian Wilson, Thermo Fisher Scientific, Tewksbury, MA USA

## Application

Microalloyed steels, often referred to as High-Strength Low-Alloy steels (HSLA), are a family of materials strengthened by the addition of "micro" alloy concentrations to low-carbon mild steel. The technology of microalloying includes elements niobium (Nb), vanadium (V) and titanium (Ti) added either singly or in combination, and sometimes in conjunction with other strengtheners like boron (B), molybdenum (Mo), nickel (Ni), chromium (Cr) and copper (Cu) to achieve desired mechanical properties. The strengthening effects of these elements make microalloyed steels particularly suited for high-strength applications by facilitating grain refinement and precipitation hardening.

The development of microalloyed steels began more than a half century ago and constitute an important category of steels estimated to be approximately 12% of total world steel production. Microalloyed steels are used in every major steel market sector in various parts of the world and their development has played an important role in the expansion of certain key industries. Some industrial applications where microalloyed steels can be found include:

- Oil & Gas line pipe
- Offshore/platforms
- Automotive industry
- Pressure vessels
- Shipbuilding/Naval vessels
- Building structures
- Bridges/suspension components
- Heavy equipment

Hot-rolled plain carbon steel is the most popular material used in construction. Its strength can be increased by raising the carbon content and is proportional to the carbon equivalent (CE), which is essentially the combined effect of the carbon and manganese content in a steel based on the formula [CE = %C + %Mn/6]. While raising the carbon content and subsequent carbon equivalent does increase strength, it also reduces other properties such as toughness, ductility and weldability. Through the strengthening mechanisms of microallying elements Nb, V and Ti, higher yield strengths can be achieved with a dramatic reduction in carbon content. This low-carbon content greatly improves weldability and

An inspection is conducted using the handheld Thermo Scientific™ Niton™ XL5 XRF analyzer.

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reduces risk of cracking for specific applications such as pipelines, bridges and buildings. Some notable advantages of using microalloyed steels are:

- Weight reduction by over 25% over hot-rolled carbon steel
- Operational savings through increased pumping capacity in line pipe
- Lower fabrication costs from weight savings in transport and handling
- Reduced carbon content improves weldability and weldment toughness
- Lower carbon equivalent (CE) minimizes heat treatment and susceptibility to cracking

In 2009 the Pipeline and Hazardous Material Safety Administration (PHMSA) issued advisory bulletin ADB-09-01 to owners and operators of natural gas pipeline and hazardous liquid pipeline systems that an integrity issued had been discovered regarding microalloyed high-grade line pipe. Tests conducted on line pipe installed in pipeline systems revealed that some of the material did not meet the requirements specified by the American Petroleum Institute (API) Specification for Line Pipe API 5L. Pipe joints produced from the same heat exhibited variable chemical and mechanical properties by as much as 15% lower than the values specified by the pipe manufacturer-produced mill test report including microalloying elements Nb, V and Ti among others.

Owner/operators need the confidence that the growing number of high pressure, large diameter hazardous liquid and natural gas pipelines are built to the highest standards and fully tested with the best available technology to comply with safety guidelines and reduce susceptibility to failure. Verification of material chemistry with portable X-ray fluorescence (XRF) is an ideal choice. The new Thermo Scientific Niton XL5 allows for fast, accurate and precise analysis of microalloying elements in steel and other elements in the field.

## Thermo Scientific Niton XL5 XRF Analyzer

The new Niton XL5 is the smallest and lightest high performance X-ray Fluorescence (XRF) metal analyzer in the market. The light weight and small size of the Niton XL5 reduces operator fatigue and enables access to more test spots. Compact measurement geometry and new powerful 5W X-ray tube provides highest performance and best sensitivity for the most demanding applications such as residual element measurement. The Niton XL5 provides the following key benefits:

- Unparalleled chemistry and metal grade identification accuracy for confident results every time
- Excellent micro element detection (Nb, V, Ti) for fast and reliable analysis of microalloyed steels
- Flexible user interface enables custom workflow solutions and easy optimization for specific application. Niton XL5 pseudo element feature can be used to automatically calculate sum of the microalloying elements.
- Integrated camera and small spot analysis for accurate sample positioning and image capture
- Waterproof, dustproof and rugged housing for harsh environments

The Thermo Scientific Niton XL5 is the latest offering in our market-leading family of Niton handheld XRF analyzers. The Niton XL5 is specifically designed for low detection limits, high accuracy, and the fastest analysis time for microalloying elements, such as Nb, V, and Ti.


Niton XL5 pseudo-element feature enables automatic calculation of the sum of microalloying element. Analyzer screens featured correlate to repeatability study results, next page: left screen to Table 1; right screen to Table 2.

## Test Method and Results

Using a handheld Thermo Scientific Niton XL5 XRF analyzer, certified reference standards and samples were analyzed after ensuring the surface is clean of any contaminants. Carbon steel alloys oxidize easily when exposed to atmospheric conditions. This oxide coating can affect the accuracy of the reading when performing an XRF analysis. It is important to remove any corrosion in order to ensure accurate reading. In addition to oxidation, there can often be paint or oil or grease on the surface. All surface contamination must be removed in the area to be analyzed.

Data quality objectives dictate the sample preparation requirements and the minimum analysis time used. Ten individual XRF readings were collected using a total measurement time of 30 seconds. Objective of the micro element analysis is to discern whether the delivered material contains the appropriate levels of microalloying elements Nb, V and Ti as certified by the corresponding Material Test Report (MTR). In addition, the sum of these elements can be verified to meet the acceptance criteria and not exceed restrictions prescribed in the material specification or purchase order.

For API 5L line pipe, the current limit on the combined microalloying content Nb+V+Ti is 0.15 percent for steels with yield strength > 60 ksi.

| Repeatability Study Results of Microalloy Elements in Carbon Steel Samples<br>Round #1 Results Using the Niton XL5 | | | | | |
|---|---|---|---|---|---|
| **Run Number** | **Nb** | **V** | **Ti** | **Nb+V** | **Nb+V+Ti** |
| **1** | .063 | .072 | .068 | .135 | .203 |
| **2** | .058 | .075 | .068 | .134 | .202 |
| **3** | .060 | .073 | .069 | .133 | .202 |
| **4** | .064 | .075 | .070 | .139 | .209 |
| **5** | .063 | .073 | .069 | .136 | .205 |
| **6** | .063 | .074 | .073 | .137 | .210 |
| **7** | .058 | .073 | .072 | .131 | .203 |
| **8** | .057 | .068 | .069 | .125 | .194 |
| **9** | .060 | .073 | .067 | .133 | .200 |
| **10** | .061 | .073 | .070 | .134 | .204 |
| **Material Test Report** | **.054** | **.073** | **.066** | **.127** | **.193** |
| **Niton XL5 Average Result** | **.061** | **.073** | **.070** | **.134** | **.203** |

| Repeatability Study of Microalloy Elements in Carbon Steel Samples<br>Round #2 Results Using the Niton XL5 | | | | | |
|---|---|---|---|---|---|
| **Run Number** | **Nb** | **V** | **Ti** | **Nb+V** | **Nb+V+Ti** |
| **1** | .012 | .043 | .010 | .055 | .065 |
| **2** | .013 | .041 | .012 | .054 | .066 |
| **3** | .016 | .042 | .009 | .058 | .067 |
| **4** | .017 | .040 | .011 | .057 | .068 |
| **5** | .015 | .046 | .010 | .061 | .071 |
| **6** | .014 | .043 | .011 | .057 | .068 |
| **7** | .012 | .044 | .010 | .056 | .066 |
| **8** | .015 | .041 | .010 | .056 | .066 |
| **9** | .016 | .042 | .010 | .058 | .068 |
| **10** | .015 | .041 | .010 | .056 | .066 |
| **Material Test Report** | **.013** | **.045** | **.010** | **.058** | **.068** |
| **Niton XL5 Average Result** | **.015** | **.042** | **.010** | **.057** | **.067** |

Table 1 and Table 2: Represent the data collected from a repeatability study conducted with carbon steel samples using the Niton XL5 analyzer. Ten individual tests were conducted for 30 seconds each in two sample rounds. The Niton XL5 Average Result is shown for each round. The one sigma standard deviation was calculated using the ten individual results from each round. As shown by the test results, agreement between laboratory results and the Niton XL5 analyzer is consistent.

| Niton XL5 Limits of Detection | | | | |
|---|---|---|---|---|
| **Measured Time, Seconds** | **Ti** | **V** | **Nb** | **SUM** |
| **15** | .0036 | .0036 | .0012 | .0084 |
| **30** | .0025 | .0025 | .0008 | .0058 |
| **60** | .0018 | .0018 | .0006 | .0042 |

Table 3: Limits of detection (LODs) for the Niton XL5 are listed in Table 3. LODs are calculated as three standard deviations (99.7% confidence interval) for each element at several different measurement times; limit of detection improves as a function of measurement time.

## Conclusion

Results achieved using the Niton XL5 analyzer demonstrates excellent agreement with the material test reports.

The Niton XL5 provides excellent trace and micro element precision and sensitivity for microalloyed steel analysis. Given appropriate sample preparation, the analyzer is able to quickly and reliably verify whether the levels of microalloying elements are in agreement with the mill test report and meet the chemical composition requirements per specification.

The success of microalloyed steels is due to complimentary strengthening mechanisms, specifically grain refinement and precipitation hardening developed from the addition of key elements niobium (Nb), vanadium (V) and titanium (Ti). Manufacturing technologies for microalloyed steels have improved rapidly over the last several decades often due to the demands from the linepipe arena in areas such as oil and gas extraction, construction and transportation.

When low detection limits or the highest sample throughput are critical, our combination of hardware, software, and direct industry experience provides the ideal solution for the most difficult analytical requirements. The improved analytical capability for trace quantities of Nb, V, and Ti and other unique capabilities make the Niton XL5 the ideal tool for micro element analysis. In addition to residual elements, the Niton XL5 can quickly provide full chemical analysis and positive material identification (PMI) for a wide range of alloy grades used in the oil and gas industry.

To discuss your particular applications and performance requirements, or to schedule an on-site demonstration and see for yourself how Thermo Scientific portable XRF analyzers can help save you time and money, please contact your local Thermo Scientific portable XRF analyzer representative or contact us directly by email at niton@thermofisher.com, or visit our website at www.thermoscientific.com/portableid.

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**Americas**
Boston, USA
+1 978 642 1132
niton@thermofisher.com

**Europe, Middle East, Africa**
Munich, Germany
+49 89 3681 380
niton.eur@thermofisher.com

**India**
Mumbai, India
+91 22 6680 3000
ininfo@thermofisher.com

**Asia Pacific**
New Territories, Hong Kong
+852 2885 4613
niton.asia@thermofisher.com

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