CHARACTERISATION OF SCALE HARDNESS

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Abstract

An in-situ scale hardness measurement is required that is repeatable and simulates the descale process. This would provide objective assessment of scale hardness, and allow correlation of scale hardness with process operating conditions, and provide improved estimates of descale turn-around times when a vessel is taken off-line.

The standard methods of measuring rock strength, Unconfined Compressive Strength (UCS) and Brazilian Tensile Strength (BTS), do not necessarily reflect the issues faced by descale crews in regard to hardness of scale. Unlike scale in process vessels where descale is undertaken by impact on a confined bulk, UCS and BTS are slow crush measurements on a small unconfined sample. Due to the structure of scale, UCS and BTS measurements often have poor repeatability and vary with orientation of the scale. Two alternative methods for measuring scale hardness are the "Schmidt Hammer" that measures the rebound of a spring loaded mass, and "Penetration Resistance" tests that measure the depth of penetration of a "pin" fired into the surface. Both of these methods more closely simulate descale by impact compared to UCS and BTS, can be performed in-situ, return results quickly, and can indicate local variations in scale hardness.

Schmidt Hammer measurements have proven to be the quickest, easiest, and most reliable hardness tests for in-situ measurement of scale hardness. Calibration of the Schmidt Hammer against standard tests such as UCS or BTS is possible. While these are technically not measuring the same properties, it does allow for crude comparison of scale to other materials such as concrete or granite, and thus selection of descale equipment with appropriate power and robustness.

Notation and units

UCS = Unconfined Compressive Strength
BTS = Brazilian Tensile Strength
MPa = Mega Pascals
mm = milli metres
A/TC = ratio of alumina to total caustic in solution

1. Introduction

Scaling of process vessels and pipework occurs throughout Bayer process alumina refineries due to contact between hot supersaturated process solutions and metal surfaces. Scale formation can be compounded by the presence of particulate material in slurries. In regions of low flow, the particulate material can settle and be cemented by the phases crystallising from solution, accelerating the overall rate of scale formation. As equipment design and operating practices are improved, it would be expected that scale in some process areas may become harder. For example in thickeners, as high mud level events become less frequent and A/TC ratios are increased, the wall scale might be expected to be composed primarily of hard and continuous gibbsite growth scale, rather than have a partial muddy composition that makes the scale less tenacious. Although the rate of scale growth is reduced, the descale effort required may increase significantly. Current evidence regarding hardness of scale and its relation to operating parameters and descale effort are largely anecdotal because a reliable quantitative measure of scale hardness is not available.

A quantitative measure of scale "hardness" would allow:

- Estimation of descale turn-around times when a vessel is taken off-line for descale
- Identification of suitable tooling to effectively descale vessels
- Correlation of scale hardness with process operating conditions to predict in advance descale turn-around times, and to determine whether operating conditions can be tuned to reduce scale formation without impacting on production.

Such a quantitative measure of hardness would ideally need to be:

- Quick to undertake
- Performed on location
- Require minimal sample/surface preparation
- Readily relate to standard methods
- Relevant to the descale issues
- Representative and statistically significant.

2. Hardness vs Toughness

In the refinery operating areas, the term "hardness" is generally used in association with scale when describing the effort required to remove scale. However, both "hardness" and "toughness" of scale contribute to the effort required during descale. Hardness and toughness are distinct properties (although overlap in terminology exists in some areas). For the purposes of this paper, the terms will be used as follows:

Hardness – Resistance of a surface to indentation or deformation under impact. Also relates to rebound of the impacting object.

Toughness – Resistance to fracture when a material is stressed.

When scale is removed using impact tools, the hardness of the scale determines the effort required to initially penetrate the scale, and the toughness determines how the scale fractures and breaks i.e. as large pieces, or small pieces, or not at all. If a scale is composed of layers with varying compositions and physical properties as is often the case, hardness and toughness characteristics are likely to vary with direction. The structure of the scale and place-to-place variations will also affect the progress of descale.

The effective toughness of the scale will also be affected by the stage of descale. In a vessel that is scaled around its entire inner circumference, the scale is confined and self supporting, and mechanical impacts are all resolved into compressive forces. Much effort is required to establish the initial break into the scale. Once a working edge has been established and the scale is no longer confined, scale removal can proceed more rapidly by breaking lumps of scale away from the edge.

3. Measuring the Hardness and Toughness of Scale

Various methods for quantifying scale hardness and toughness have been considered and appraised against the requirements indicated previously. An overview is given in Table 1, and more detail provided in the paragraphs that follow.

3.1 Unconfined Compressive Strength (UCS) and Brazilian Tensile Strength (BTS)

In both the UCS and BTS tests, a cylinder of the test material is obtained by coring, and cut to obtain a specified length/diameter ratio. In the UCS test, force is applied along the axis of the cylinder, and in the BTS test force is applied across the diameter of the cylinder (Figure 1). In each case the force is progressively increased until the sample integrity fails.

These measurements are used widely in the materials, mining and building industries, and various standard procedures exist. Descale tooling requirements could be selected by comparison to a particular rock type or grade of concrete for example. However, the process from obtaining a sample through to completion of the measurement requires considerable time and effort, limiting the number of samples that can be collected and analysed for repeatability, and preventing rapid feedback for estimation of the time required to descale a vessel. Furthermore, the tests are undertaken by a progressive crushing force on an unconfined sample, and do not simulate the high impact of mechanical descale, the most difficult part being the initial cut into confined scale.

While these measurements can be used for comparison of scale strength to other materials, they are not suitable for rapid feedback of results and statistical representation of variations, and have limited relevance to the actual descale process.

3.2 Schmidt Hammer

The Schmidt Hammer (Figure 2) measures the rebound of a spring loaded mass impacting against the surface of the sample. It is simple to use, results are returned immediately and multiple measurements can be made quickly providing good statistics and place-to-place variations. The measurement can be calibrated against standard samples, and indicates surface hardness (not toughness) albeit at a significantly lower impact energy than occurs during descale. The instrument can compensate for direction of the measured surface (vertical, horizontal, or angle), but requires the surface to be flat, smooth and free of surface particulates. If these requirements are not met, the measured result can be significantly in error.

While having some limitations, the Schmidt hammer can be used to quickly obtain measurements of scale surface hardness, with indication of place to place variations.

3.3 Penetration test

In the penetration test, a small explosive charge in a hand tool is used to impact a weight onto a “pin”, driving it into the surface of the scale. The penetration of the pin is measured. The method is relatively easy to perform, results are obtained quickly and multiple measurements can readily be made providing good statistics and place-to-place variations. The method can be calibrated against other methods, and probably provides a combination of information about surface hardness, and toughness over a very localised area. It is expected that would be some limited relevance to the larger size scale toughness encountered during descale. The surface needs to be flat and smooth to allow accurate measurement of the pin penetration (Figure 3).

While having some limitations, the penetration test can be used to quickly obtain indications of scale surface hardness and toughness, with indication of place to place variations.

3.4 Fracture toughness tests

Various configurations of fracture toughness tests are available, but all essentially rely on supporting a sample on multiple rails or points, and applying a load that is progressively increased until the sample breaks (Figure 4). A direct measure of toughness is obtained. The sample size that can be analysed depends on

Table 1. Overview of benefits and limitations of various methods for quantifying scale hardness and toughness

	Speed/ease of measurement	On location	Standard method	Relevance to descale	Representative of whole
UCS/BTS - Unconfined Compressive Strength / Brazilian Tensile Strength	Long turn-around time for results. Core samples to be extracted and prepared.	Ex-situ measurement at external laboratory.	Yes – used in materials, mining and building.	Low relevance. Crush measurement on unconfined sample.	Limited representation. Sample preparation limits number of measurements.
Schmidt Hammer	Rapid data collection. Easy to use.	In-situ.	Can be calibrated to standard methods.	Simulates impact of mechanical descale tool on scale surface, but at significantly lower energy.	Rapid in-situ data collection allows statistically representative data for surface hardness.
Penetration test	Reasonably quick and easy to use.	In-situ.	May be calibrated to standard methods.	Simulates impact and penetration of mechanical descale tool on scale surface, but at significantly lower energy and limited extent.	Rapid in-situ data collection allows statistically representative data for surface hardness and toughness.
Fracture toughness tests	Long turn-around time for results. Samples to be extracted and prepared.	Ex-situ measurement at laboratory.	Yes – used in materials, mining and building.	Simulates fracture creation and propagation, but over limited extent.	Limited representation. Sample preparation limits number of measurements.
In-situ sensor on descale tool	Rapid collection of data during descale.	In-situ.	Non-standard measurement but calibration may be possible. Significant design and development.	Directly responds to hardness and toughness during descale.	Totally and directly representative of surface and bulk of scale, with complete statistical representation.

the equipment available. The limitation of this method is the time and effort for obtaining a suitably sized sample through to completion of the measurement. This limits the number of samples that can be collected and analysed for repeatability, and prevents rapid feedback for estimation of the time required to descale a vessel.

Fracture toughness measurements can be used for measurement of scale toughness, but are not suitable for rapid feedback of results and statistical representation of variations.

3.5 In-situ sensor on descale tool

The ultimate method of measuring scale hardness and toughness would be to have a sensor or transducer mounted on a mechanical descale tool to directly and continuously measure the response of the tool during impacts on scale during descale activities e.g. a strain sensor, or accelerometer. This would not directly measure either hardness or toughness of scale, but with experience data could probably be interpreted to represent either. The data collected would be absolutely relevant to the descale process, instantly available, and readily accumulated for analysis later (e.g. statistical analysis for overall "hardness" and "toughness", as well as variability through the depth of the scale and at various locations around a vessel). Descale operators could be provided with continuous graphical feedback of the measured parameters, and could learn to recognise patterns of amplitude and pulse shape that indicate degree of hardness, allowing them to search the scale surface for the best location to penetrate the scale. Likewise, toughness indicators would allow operators to decide whether a particular location of scale penetration was worth pursuing in regard to propagating fractures that would lead to breakage of the scale.

Clearly there would be significant engineering and logistical challenges in developing such a system. The sensor would need to be extremely robust in the physical and chemical environment that it would be operating in. Large volumes of data would have to be transmitted from the sensor and displayed continuously, as well as being recorded for later processing. Interpretation of pulse amplitudes, shapes and patterns into corresponding hardness and toughness representations may or may not present a significant challenge. The continuously collected data from the sensor would also have to be associated with the progress of descale, in regard to the location in the vessel, and position within the depth of the scale. These issues are all resolvable, but the cost may make the concept impractical to implement.

4. Sample preparation

Both of the most practical and relevant hardness/toughness tests, the Schmidt Hammer and Penetration test, require a flat and smooth surface for accurate measurements of scale hardness. The effect of sample preparation was appraised by comparing results obtained from the surface of a scale sample as received, with the results when the surface had been ground smooth. The data in Table 2 indicate that both the Schmidt Hammer and Penetration test return a lower result for hardness on the unprepared surface compared to a surface that has been ground flat and smooth (note that in the penetration

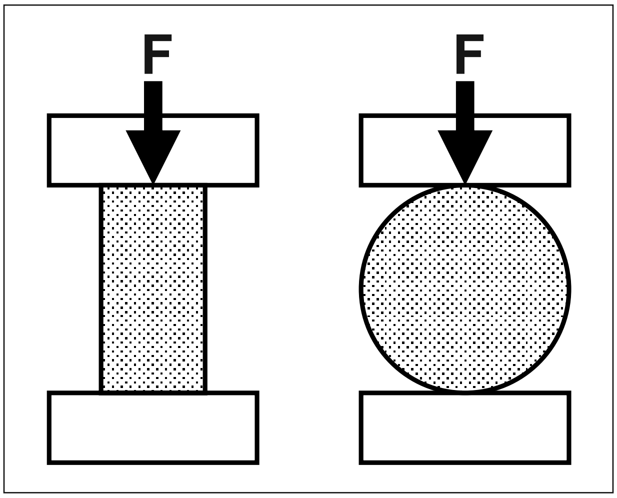

Figure 1. UCS (left) and BTS (right) tests

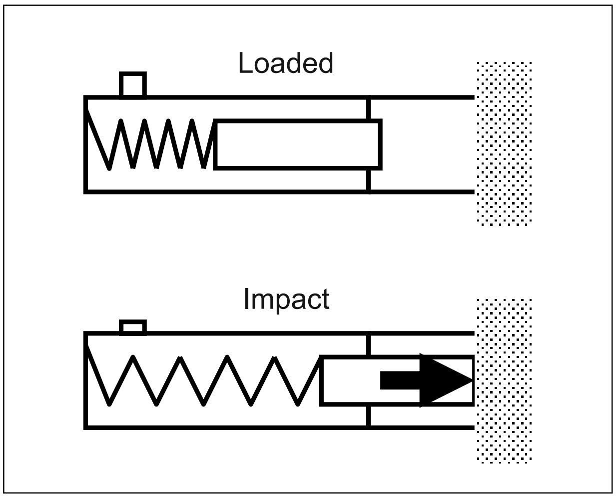

Figure 2. Schmidt Hammer concept

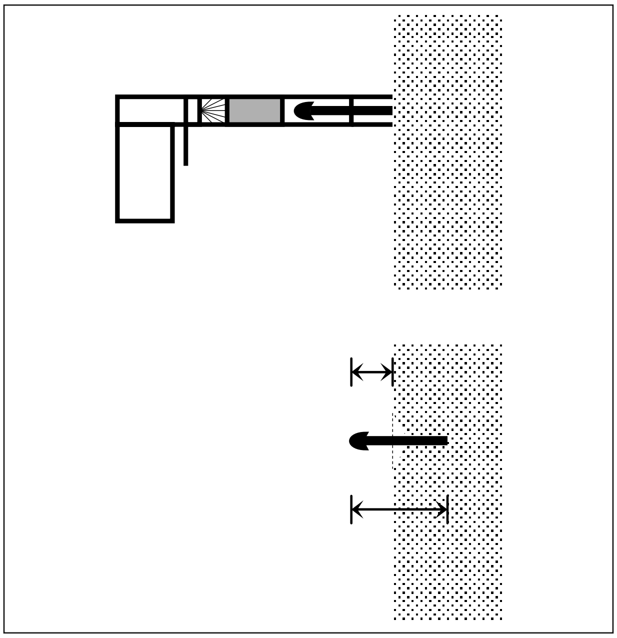

Figure 3. Penetration test concept

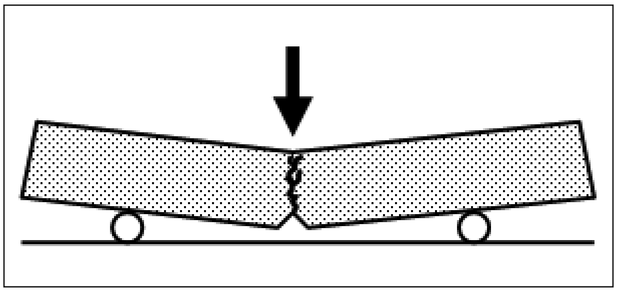

Figure 4. Fracture toughness test

test, less penetration indicates a harder surface). These are preliminary results and further data are required to confirm these observations.

Table 2. Hardness measurements vs sample preparation

	Unprepared surface	Surface ground flat and smooth
Schmidt Hammer (MPa)	30	41
Penetration test (mm penetration)	31	26

5. Hardness of scale vs drying time

Descale operators have reported that the longer the delay between a vessel being taken off-line and the commencement of descale, the harder the scale becomes, presumably due to drying of entrained liquor and cementing as dissolved solids crystallise. Measurements using the Schmidt Hammer on a sample of scale 3 months after it was collected support these observations. The hardness of the surface of the scale was measured at 41 MPa. The surface was then ground to remove scale to a depth of 5 mm, where some areas of entrained liquor were evident. At a depth of 5 mm, the dry area was measured at 40 MPa, and wet areas at 38 MPa. A further 10 mm of scale were removed by grinding, and at this depth of 15 mm below the original surface dry areas measured at 40 MPa, and wet areas at 30 MPa. The ground surface at 15 mm depth was then allowed to dry for 2 weeks, at which time the hardness was measured at 38 MPa. These limited data suggest that as expected, scale does harden as it dries, and would support a case for giving priority to descale activities after a vessel is taken off-line to reduce the descale effort required.

6. Comparison of results

In the short term, the two most practical methods of obtaining scale hardness and toughness data are the Schmidt Hammer and Penetration test. At the time of writing, there is insufficient data to compare these methods with the more standard method of UCS. While a correlation with the more standard and widely used UCS would be of some benefit, it must be remembered that these three methods are each measuring different aspects of hardness and toughness, and the results may not necessarily correlate – materials that are hard are not necessarily tough (e.g. glass is hard but brittle), and tough materials are not necessarily hard (e.g. polypropylene is tough but soft).

7. Conclusions

In-situ quantitative measurement of scale hardness and/or toughness in process vessels is complicated by a number factors including:

- Selection of a suitable and relevant test
- Anisotropy and place-to-place variation in scale properties
- Sample/surface preparation
- Turn-around time to obtain results.

The Schmidt Hammer and Penetration tests can be used as a guide to hardness/toughness with the benefits of rapid return of results and ease of collection of data to indicate local variability, but the limited relevance to hardness and toughness issues encountered during actual mechanical descale may be a shortcoming.

A truly in-situ and relevant measure of scale hardness obtained using a transducer mounted on a mechanical descale tool would require significant cost and effort to develop.

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