PARTICLE SIZE MEASUREMENT AND STIRRED MILLING TESTWORK: THE MICRON-SCALED PITFALLS WITH POWERFUL CONSEQUENCES

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ABSTRACT

In standard stirred mill laboratory testwork, the particle sizing method employed and how it affects the reported specific energy requirements are often overlooked. Lasersizing is often employed as the industry gold standard for its ability to achieve consistent and accurate results across several different commodities. However, in the context of stirred milling, particle characteristics like shape and surface properties can compromise its ability to accurately portray the sample's particle size. The underlying impact of this can be detrimental to the resulting mill sizing causing overestimation of reported power requirements. Using the IsaMill™ as a case study, this paper demonstrates why it is important to understand how the particle sizing technique selected can influence signature plot results, when to apply alternate analytical particle sizing techniques and how to improve lasersizing for signature plot testwork.

1.0 INTRODUCTION

The rise in fine grained complex ore bodies has made regrinding an essential requirement in most mineral processing operations. To achieve sufficient liberation of minerals, ultrafine grind sizes as low as 5um are now being targeted with specialised grinding technologies. To allow for these grinding technologies to scale up accurately, specialised laboratory testwork procedures have been developed for each technology to allow for scaleup. Although when it comes to particle size analysis, the gold standard has typically been associated with the use of lasersizers or screens. The problem with this is that without understanding the intricacies of the method, biases will be introduced into your stirred milling testwork due to sizing techniques. This paper aims to shed some light on some of the key attributes that need to be understood when interpreting particle size in stirred milling.

2.0 SIGNATURE PLOT

The IsaMil™ Signature Plot is the 1:1 scaleup testwork for the IsaMill™. The test allows for a 1:1 scaleup to full-scale operation through the replication of consistent laboratory and full-scale conditions such as: media size, media SG and feed density (%solids). The test procedure follows a semi-continuous batch pendulum arrangement that utilises a sample solids volume 3-4 times the active grinding volume to achieve steady state. Figure 1 shows an example of a pass through the mill, a key aspect of this test is that it is not possible to short circuit the feed and there is no potential for a particle to be ground multiple times over in a pass.

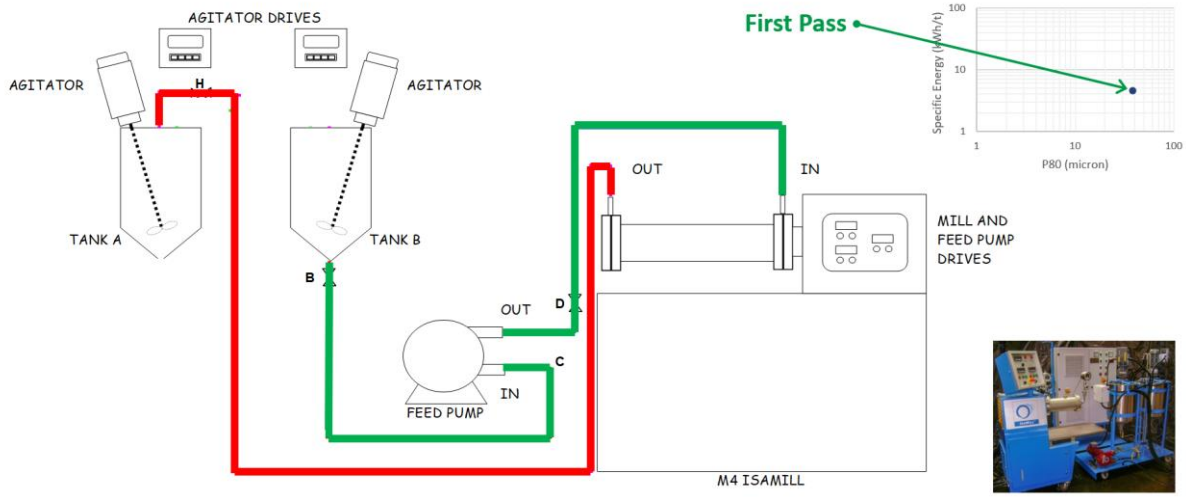

Figure 1: IsaMill™ M4 Signature plot layout (Glencore Technology, 2022)

Testwork is currently completed by accredited M4 laboratories worldwide, operating test conditions are nominated by Glencore Technology to suit the feed/product conditions and the results are reviewed as part of Glencore Technology's quality assurance and quality control process. All technicians must pass the accreditation test to verify procedural competency and mechanical performance of the lab scale mill, the aim of this is to accredit the operator, not the laboratory, to ensure consistency in the method. This accreditation is renewed every 2 years to ensure that standards are upheld and 1:1 scaleup can be achieved. According to M. Larson et al. (2012), the signature plot test will have an average margin of error of +/-5% under ideal conditions. Interestingly, the most common sources of error observed in signature plot tests and failed accreditation attempts have been due to lasersizing. Hence the inspiration for preparing this paper.

3.0 LASER DIFFRACTION PARTICLE SIZING FUNDAMENTALS

Imagine you are asked to measure the size of the irregular particle in Figure 2 (the one on the left) using one-unique number for characterisation. You measure the dimensions and find that it is 20µm x 100µm. Now describe that shape with a single number. The only shape that can be described by one unique number is a sphere (Malvern, 2014), which would be 39µm (the one on the right in Figure 2). This is what laser sizers do, and it is this fundamental approach to particle size characterisation that is the discrepancy you see between screen sizings and

laser sizings.

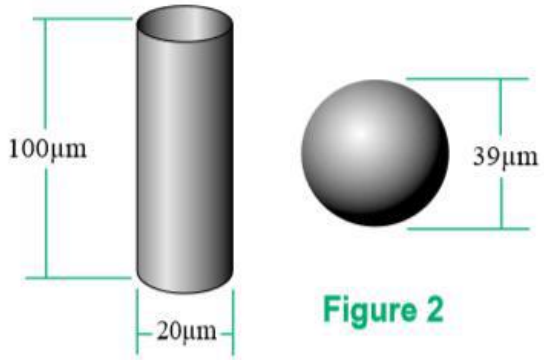

Figure 2: Volume Equivalent Sphere (Malvern, 2014)

So what really is laser sizing? Laser sizing is a well-established particle measurement technique that uses the concept of laser diffraction. Laser diffraction measures particle size distributions by measuring the angular variation in the intensity of light scattered as a laser beam passes through a dispersed particulate sample. Large particles scatter light at smaller angles and higher intensity while small particles scatter light at larger angles (weaker intensity), as seen in Figure 2. The scattering intensity data is then analysed to calculate the size of the particles responsible for creating the scattering pattern.The particle size is reported as a volume equivalent sphere diameter, which allows you to develop your particle size distribution (Malvern, 2014).

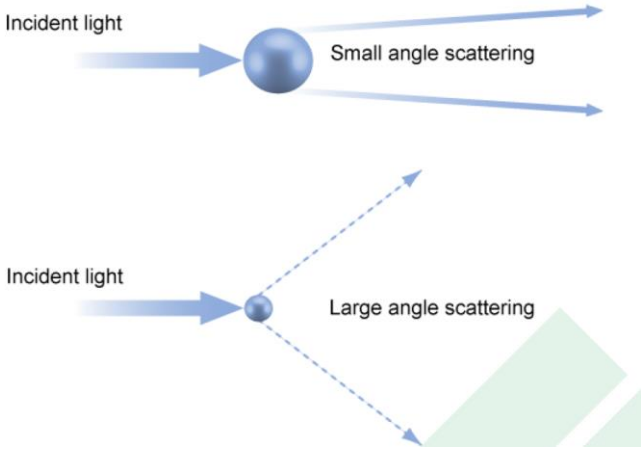

Figure 3: Light Scattering of Particles (Malvern, 2014)

Since the particle size distribution is reported as a volume equivalent sphere diameter, there are two methods that can be utilised:

- Mie Theory of light scattering is the most common and recommended method that laser sizers use. Mie theory requires knowledge of the optical properties (refractive index and imaginary component) of both the sample being measured, along with the refractive index of the dispersant.
- The other is the Fraunhofer approximation, which does not require knowledge of the optical properties of the sample. This can provide accurate results for large particles. However, it should be used with caution whenever working with samples that might have particles below 50µm or where the particles are relatively transparent. (Malvern, 2014)

4.0 LASERSIZING VS SCREENING

Screening is the most universally accepted particle sizing technique known for its robustness across a wide variety of applications. It is accomplished by passing a known weight of sample material through successively finer sieves and weighing the amount collected on each sieve to determine a proportion of weight in each size fraction (Gilroy, 2016). The physical nature of this technique means that irregularly shaped particles can pass through the nominal aperture size if screening is conducted for a long-enough duration, when favourable orientation is achieved. This can make repeatability in results challenging to achieve in laboratory conditions and strict standards for procedures must be adhered to.

Screening can also become complicated with large volumes of near-sized particles onto the apertures selected for analysis. If the sample is large enough, near-sized particles can cause blinding of finer apertures, creating a coarser size distribution. This effect becomes more prevalent in screen sizes sub 38um which is typically where another characterisation method should be recommended (Gilroy, 2016). Clay containing minerals can also agglomerate together, preventing particles from passing through the apertures.

Sample mass used can influence the effectiveness of this sizing method. Much like blinding, too much sample can accumulate over a particular sieve size creating a bed of solids. If this bed is too deep, it will minimise the probability for nominal particles to pass through that sieve (Gilroy, 2016). On the other hand, the sample mass must be large enough to adequately represent the bulk material (Gy's Sampling theory).

In most stirred milling tests, laser sizing is generally recommended as the gold standard. The technique provides consistency in readings and ease of operation as there is little preparatory work required. It is also capable of measuring particle sizes sub 19um making it attractive for regrind testwork which has traditionally targeted the ultrafine grinding duties.

One key differentiator between the two methods is the size range that can be measured. In coarser applications, achieving nominal wet dispersion in a lasersizer can be challenging as segregation of coarse particles inside the beaker can occur. Furthermore, biases can be introduced to the measurement depending on the subsampling method. For this reason, screening is typically seen as more robust when analysing size distributions coarser than 200um.

Screening also doesn't assume that particles are spherical like the lasersizer does, meaning that results cannot be directly compared to eachother. This is why it is preferable to measure particle size across a single distribution with a singular method instead of combining two results.

5.0 COMPARING APPLES TO ORANGES IN A SIGNATURE PLOT

In a minerals processing plant, it is impossible to guarantee that the particle shape is going to be spherical. Different minerals have vastly different crystal structures. For example, galena has a cubic structure while micaceous silicates are thin and platy (Figure 4). The effect of crystal structure on the analytical technique can lead to some significant differences in results on a signature plot.

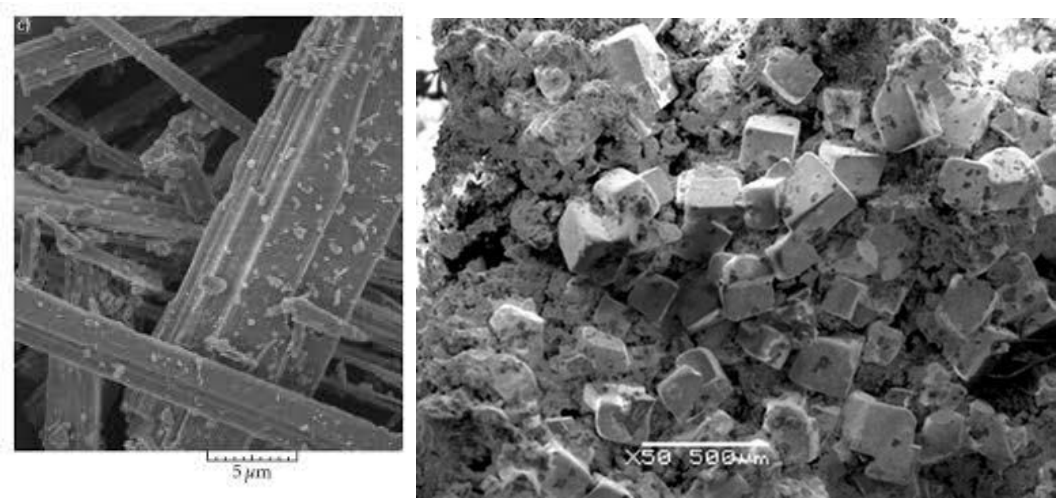

Figure 4: Mica SEM (left) - (Kovalcik, Machovsky, & Wimmer, 2012) and Galena SEM (right) - (Russo & Campostrini, 2008)

5.1 How does this influence my flowsheet?

The effect of crystalline structure and its influence on the signature plot is clearly displayed (Figure 5). A tertiary milling feed was concurrently analysed by both screens and a laser sizer. When the screens were utilised a reported 30% reduction in specific energy across all size fractions could be seen. If the mineralogy wasn't understood prior to the test, and if different grinding tests were undertaken with different particle characterisation methods, this improvement could be mistaken for "grinding efficiency".

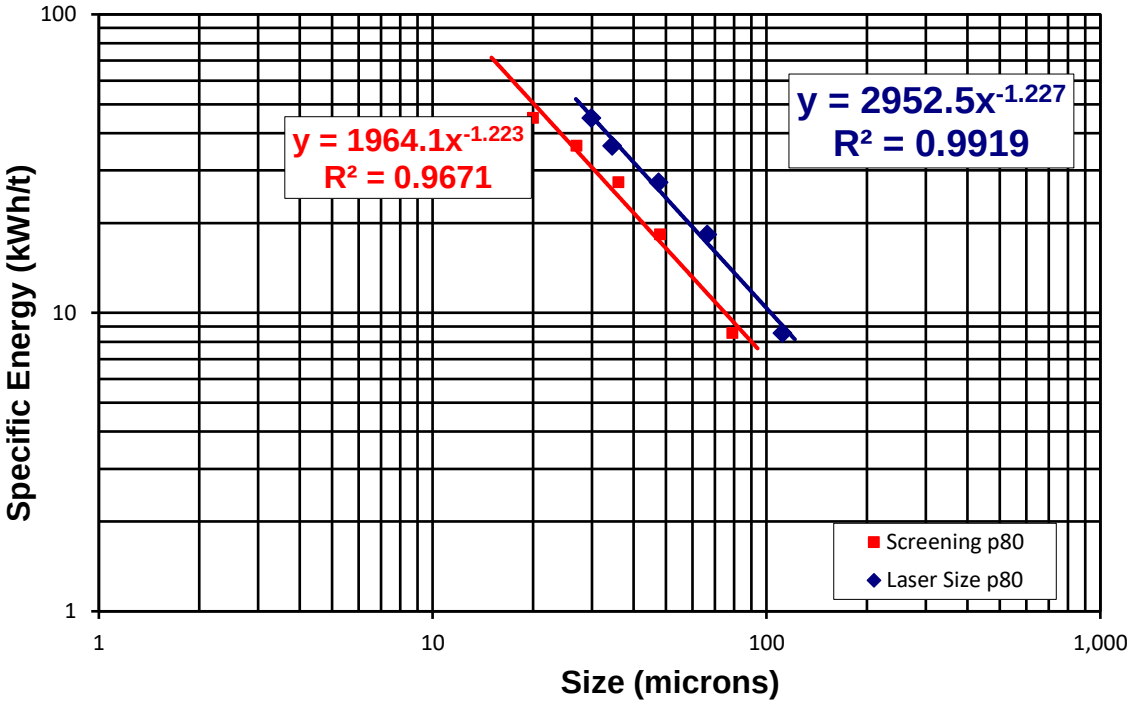

Figure 5: Discrepancy between laser sizing and screens – Tertiary Copper Grinding (Gurnett & Johnston, 2025)

The reason is due to a fundamental difference in how size is characterised in both methods. In screening, particles must physically pass through sequentially reducing aperture sizes. If a particle is captured on a screen, it is reported as coarser than the aperture size, while if it passes through, it is finer. Because of the physical nature of this characterisation technique, circumstances exist where a particle can self-orientate and pass through a screen.

Particle size analysis on a screen is therefore determined by both shape and particle size. In a stirred milling test this gives the illusion of an improvement in energy efficiency

Another example of this can be seen in Figure 6 where there was a reported reduction in specific energy on a Copper rougher concentrate.

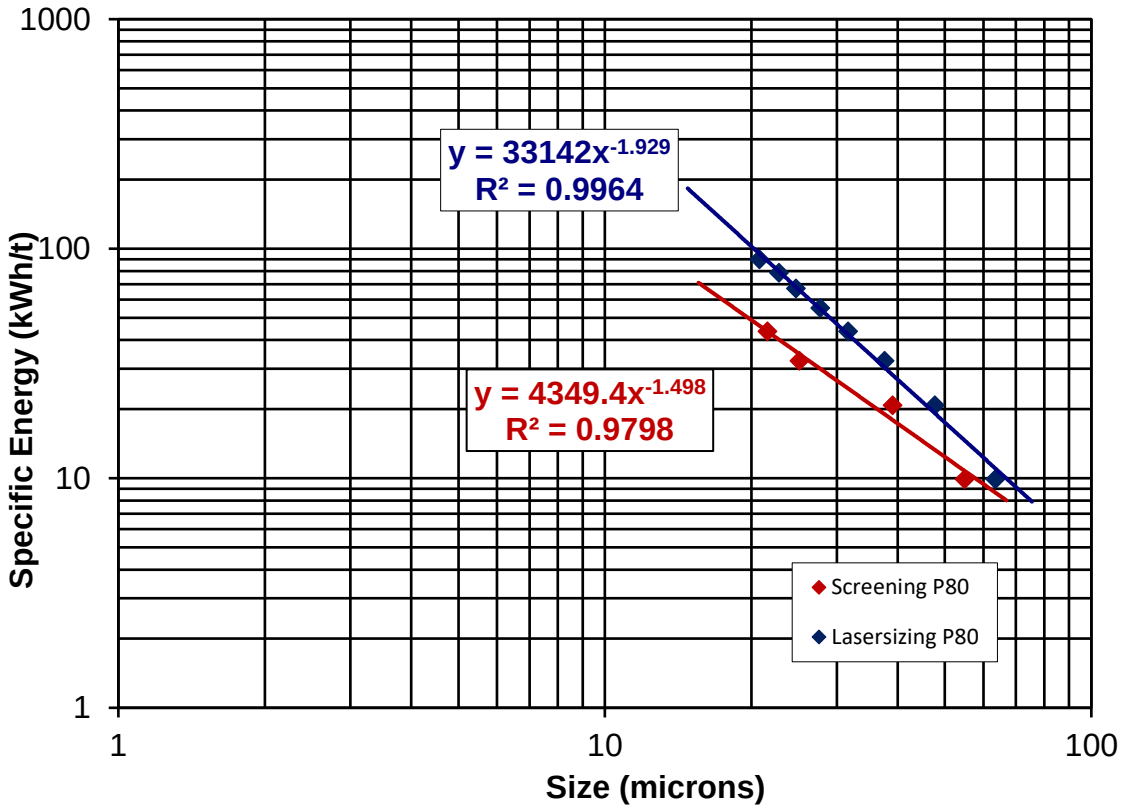

Figure 6: Copper Rougher Concentrate - signature plot lasersizing vs screens

In this result, screen sizings showed a reported decrease in specific energy from 23% to 48% as the P80 became finer. If this was taken to a real world example, where a 70tph IsaMill™ was sized targeting a 30um P80 grind as a case study, an M15,000 (3.8MW) in closed circuit would be recommended from the lasersizing results. Alternatively, if the same mill was to be sized off screens, an M7,500 (2.2 MW) in open circuit would have been recommended, highlighting the real world significance of sizing characterisation methods on the mill sizing (Table 1).

Table 1: IsaMill™ sizing case study as a result of sizing methodology

Analytical Method	Screen Sizings	Lasersizing
Specific energy to P80 30um	27.4 kWh/t	47.4 kWh/t
Estimated Drawn Power	1890 kW	3318 kW
Mill size recommended	M7,500 (open circuit)	M15,000 (closed circuit)

5.2 So what do I need to take away?

How does this apply to anyone else in the industry? This conundrum does look like a procedural problem that the vendor/lab should rectify. However, if you find that you are doing technology trade-offs between regrind testwork, it may be worth delving a little deeper into the results.

When conducting a technology trade-off between regrind mills from scaleup testwork, Metallurgists must consider whether they are comparing apples to oranges. If one testing method has used screens (ie Bond work index test), then all comparative regrind tests must also use screens where possible. Failure to achieve consistency across methods can show "significant improvements in grinding efficiency" that don't actually exist – an unfair test.

Furthermore, if completing flotation characterisation testwork, it is important to understand that if screens have

determined the optimum grind size on a grade-recovery curve, then using a lasersizer in stirred milling testwork can potentially create a significant over bias in specific energy – requiring more energy than necessary to achieve sufficient liberation. Once a particle sizing method has been selected, all subsequent tests must use the same sizing procedure to minimise biases.

6.0 SIZE MATTERS, SO WHAT RED FLAGS SHOULD BE SCRUTINISED?

6.1 Mineralogy

Choosing between lasersizing and screens can have powerful consequences on the specific energy in scaleup testwork. Understanding the mineralogy of the sample and gangue minerals in testwork can assist with determining whether shape factors or other surface properties may impact results. Table 2 below has been prepared to raise awareness of some minerals where problems in sizing techniques have arisen in IsaMill™ signature plot testwork. If large quantities of problematic minerals are present, you could see issues with your size analysis.

Table 2: Known Problematic Minerals

Mineral	Viscosity effects	Shape Factors	Agglomeration	Sizing Method Recommended
Barite	☑			Lasersizing
Talc / Serpentine	☑	☑		Screening alongside lasersizing
Mica		☑		Screening alongside lasersizing
Clays	☑		☑	Screening alongside lasersizing
Magnetite			☑	Screening only
Muscovite	☑	☑		Screening alongside lasersizing
Graphite		☑		Understanding of material properties required. Target power instead of grind size.

6.2 Particle size distribution

Sometimes the particle size distribution can provide significant insights into whether agglomerates and shape factors exist. Simply looking at a cumulative size distribution can hide key information for diagnosis, so the full-unaltered size distribution needs to be considered.

A key identifier to interrogate in stirred milling testwork is the P98 product size and the ratio of P98/P80 –the coarse size index (CSI). Figure 7 shows a gold rougher scavenger signature plot with an increasing CSI and a steeper P98 curve than the P80. Typically with IsaMill™ testwork it is expected that the CSI decreases and converges to a value of 2 as the grind becomes finer. Upon initial inspection:

- Marsh Funnel viscosity readings remained constant between passes – Beads retained consistent fluidisation
- Net Power drawn remained relatively consistent between passes
- Feed size distribution was tight with a P80 of 60um and P98 of 136um (CSI 2.2)
- Particle size distributions throughout each pass were not bimodal
- Media selection was on spec at 3mm with typical GT recommendations

With testwork factors considered, a trend like this can highlight the presence of either shape factors or agglomerates. In this example, shape factors were identified with the feed size (F80) reporting 60um on lasersizer and 50um on screens.

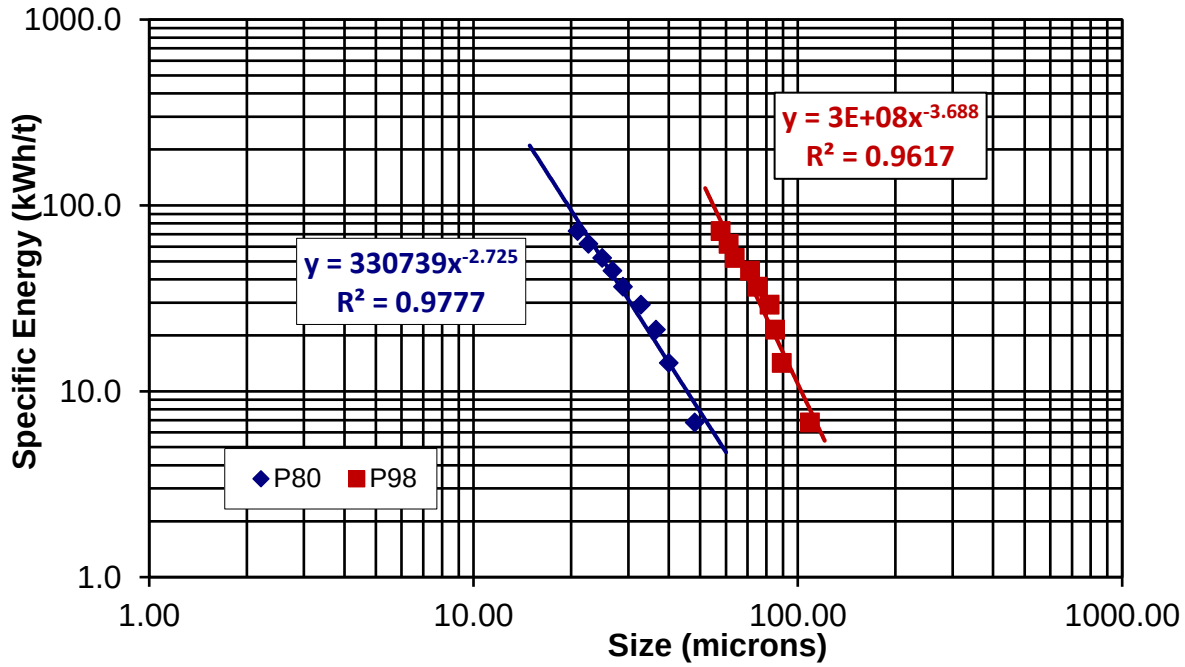

Figure 7: Gold Rougher Concentrate – P98 trend

6.3 Morphologies

Morphology of crystals can also be used to determine if a shape factor bias may be present in results. If this technology is available, it can be useful for understanding differences in results. Figure 8 and Figure 9 show the morphologies at different screen sizes from a sample taken in pass 3 of a copper signature plot. There is a significant presence of particles with large aspect ratios across each of the size distributions. The lasersizing P80 was reported as 37.6um and the screening P80 was reported as 25.1um which did lead to a significant difference between specific energy results.

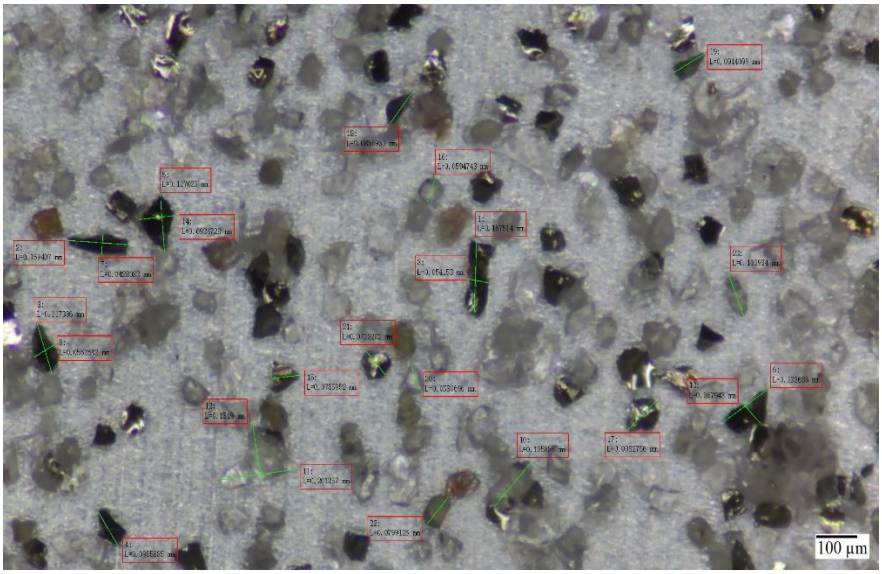

Figure 8: Sample Morphology at 45um - Pass 3 - Copper sample

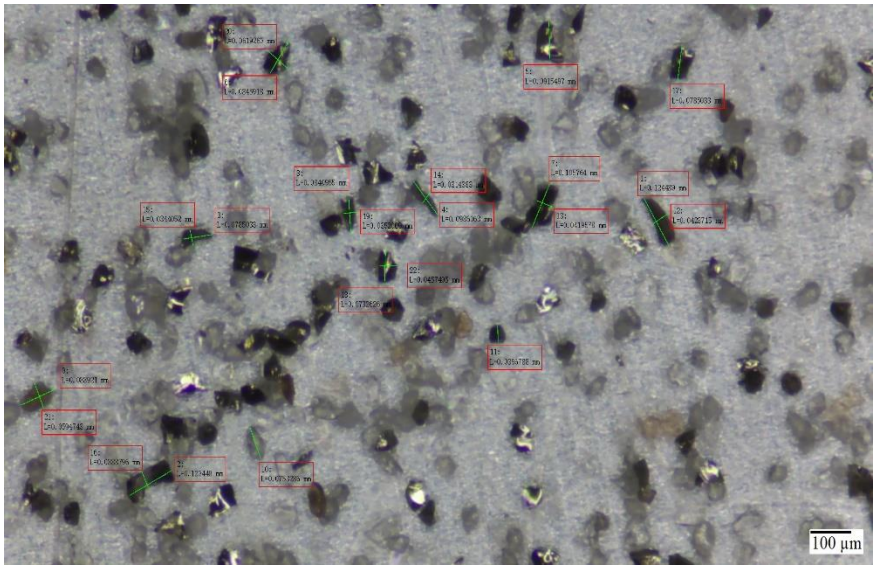

Figure 9: Sample Morphology at 37um - Pass 3 - Copper sample

6.4 Poor Regressions

The lead rougher concentrate example in Figure 10 is another demonstration of the red flag for particle size distribution. The CSI ratio increased up to 9 from an initial 2.4 in the feed. The red flag in this example was a low R^2 of 0.64 in the P98 curve indicating a poor data fit to the regression curve. Shape factors in this sample were identified and after screening this result, pass 1 dropped from a P80 of 32um to below 19um. Using the lasersizer, specific energy to achieve the same grind size was overestimated by >65%.

This is a very good example of being aware of the particle sizing methods, as an engineering company has published this as an example of the IsaMill™ not being able to grind properly and the result ofcoarse particle build up. When simply the particle characterisation method was not properly selected in the analysis of their results. As a rule of thumb when analysing signature plots, tests that achieve R^2 values below 0.90 in either P80 or P98 trendlines should be scrutinised and not accepted as is.

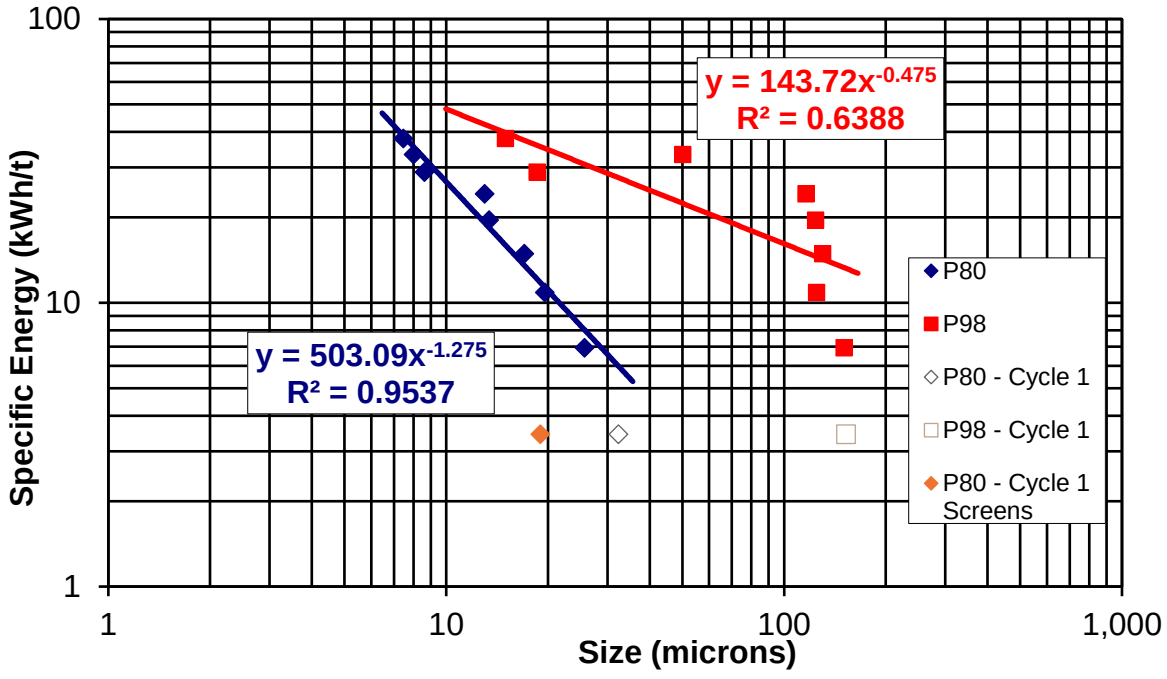

Figure 10 - Lead Rougher Concentrate – Regressions

6.5 Agglomerates

Agglomeration is a commonly observed phenomenon with mineralised slurries. Achieving good dispersion is key to ensuring accuracy in scaleup testwork, especially when dealing with finer particle sizes. Agglomerates can form from a variety of reasons such as surface charge, steric effects or even magnetisation as observed in magnetite duties. The level of inter-particle attraction can also change with grind size making it challenging to manage consistently.

Figure 11 shows an example of agglomerations which has affected the particle size distribution. Two samples from pass 1 of a signature plot were analysed in a lasersizer. Each sample underwent 2 rounds of lasersizing – the first without ultrasonics and the second with ultrasonics to break down agglomerates. The highlighted trends show the effect of ultrasonics on the particle size distribution (PSD) while the un-highlighted trends showed a distinct "tail" with a shorter peak (undispersed agglomerates). The extra volume of particles detected in the +100um region inflates the P98 value and overestimates the P80 figures reported.

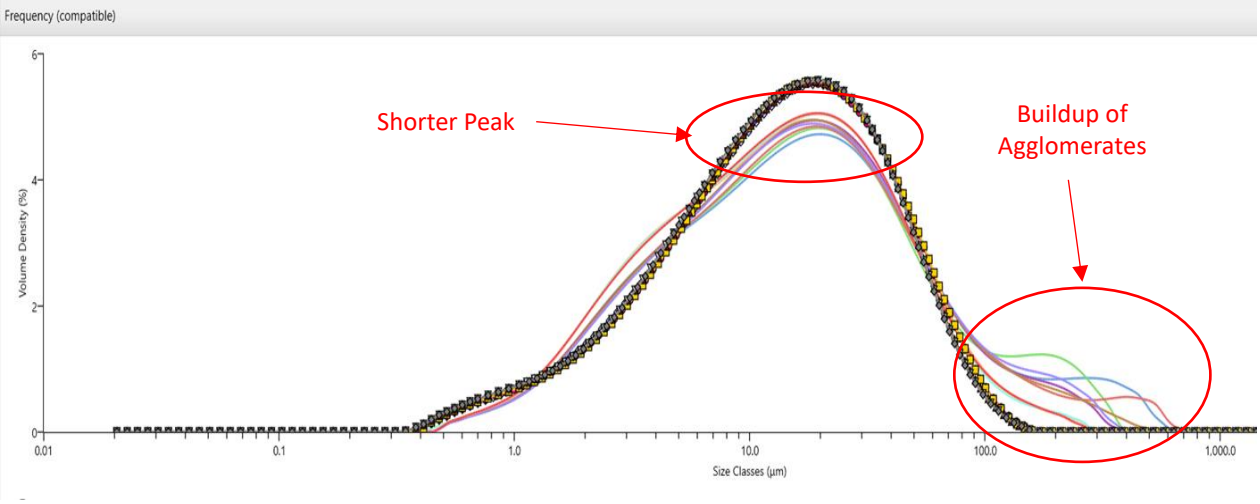

Figure 11: Agglomerations in Pass 1 of a Copper Signature plot

One of the tell-tale signs of agglomerate build-up are changes to laser obscuration readings throughout the measurement (the "cloudiness" of a sample from the surface area it consumes). Decreasing obscuration readings can indicate agglomerates are forming as fines turn to coarse and the light scattering becomes more intense. A good measurement should achieve the following:

- Consistent repeatability across measurements for D10, D50, D80 and D90 – results overlay demonstrating repeatability
- Consistent obscuration readings - no obvious trends means that the surface area of a sample is consistent meaning that particle size reported is not changing.

If agglomerates are detected, ultrasonics can be used to assist with dispersion. However, this does not solve all issues and sometimes ultrasonics can exacerbate the problem. In such cases the sample preparation procedure requires rigorous attention and even then this does not fully solve problems encountered from steric effects and surface charge.

6.6 Sample Method

Sampling method has an effect on the consistency in results and accuracy of the test. Collected samples from a signature plot should be mixed in a baffled beaker with an overhead stirrer. Depending on the ore, mixing in a stirrer for more than 2 minutes can also assist with breaking down agglomerates. Subsampling should taken from the centre of the beaker, in between the baffles to ensure a representative sub-sample is collected in a pipette (Glencore Technology, 2024). Other forms of sample preparation such as coning and quartering a dried sample can introduce too much inconsistency/error and are not recommended.

Subsample volume drawn up through a pipette should be completely emptied into the dispersion unit of the lasersizer. Segregation of coarse particles inside the bulb of the pipette can occur and adding partial volumes of subsample into the lasersizer can introduce biases to results (Larson, Anderson, Barns, & Villadolid, 2012).

Furthermore, sub-samples should be added to the unit quickly to minimise re-agglomeration in the pipette before measurement.

Too much sub-sample volume for measurement can introduce multiple scattering. The effect is more common in ultrafine duties, where a large number of particles cloud the measuring system, returning a finer particle size (Malvern, 2014). Best practice is to ensure that obscuration is kept as close to 10% as possible to minimise bias caused by this effect (Glencore Technology, 2024).

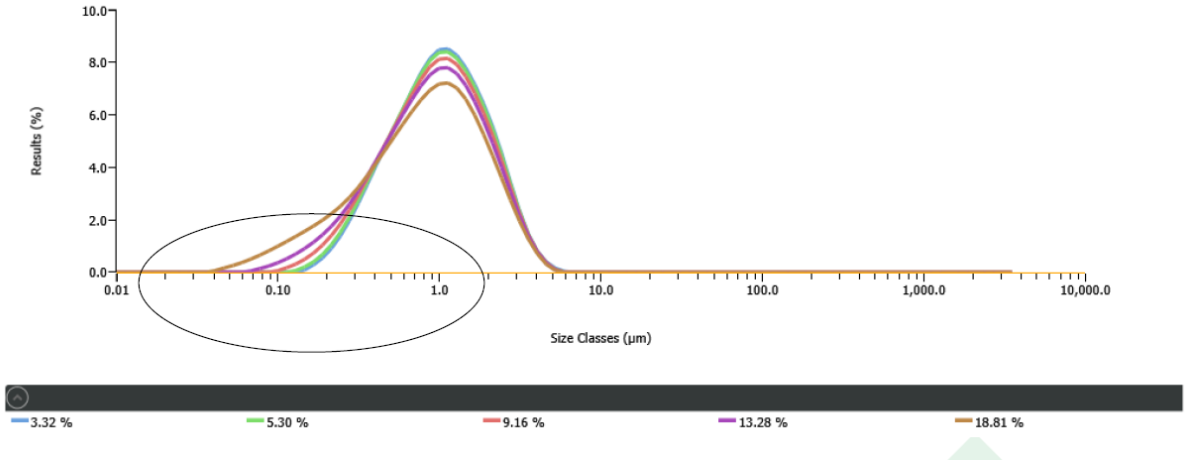

Figure 12: Obscuration Titration of the same sample (Malvern, 2014)

6.7 Faulty Laser Sizer

One of the main hurdles in passing the IsaMill™ M4 accreditation, is due to the condition of the laser sizer. The IsaMill™ Signature Plot procedure can take up a significant amount of time in preparation, testing and clean-up, so to run the samples that have been collected in a faulty lasersizer is effectively invalidating all the efforts in the equipment testing.

Table 3 is an example from a lab that was undergoing an accreditation process; it shows the difference in specific energy between a recently calibrated and serviced laser sizer against a faulty laser sizer. Duplicate samples were taken from the same signature plot and tested before and after the service/calibration. GT accepts a small error in IsaMill™ signature plot testwork and anything outside this margin is deemed inaccurate. If an IsaMill™ were to be sized from this test, it could potentially be undersized by up to 30%.

It is always important to ensure that calibrations are up to date and that equipment quality is maintained through frequent servicing. Neglecting equipment can cause significant errors in specific energy performance determination that can have very expensive real-world consequences.

Table 3: % Error in Specific energy to P80 Grind size on a standardised sample

P80 Grind Target	35um	30um	25um	20um
Test 1 – Faulty lasersizer	-10%	-16%	-24%	-32%
Test 1 – After Service	9%	6%	2%	-2%

6.0 CONCLUSION

This paper aimed to shed light on the challenges of achieving accuracy on stirred milling testwork. There is no one sizing technique to rule them all and the lasersizer has been proven as a robust solution for regrind testwork with its own unique challenges. It is hoped that the effect of particle shape and agglomerates are better understood when analysing testwork, and that for true consistency, apples-to-apples comparisons are made where possible to minimise bias in benchmarking exercises and poor reporting of results.

7.0 REFERENCES

Gilroy, T. (2016). Particle Size Analysis. In B. Wills, & J. Finch, *Wills' Mineral Processing Technology* (pp. 91-107). Cornwall: Butterworth-Heinemann.

Glencore Technology. (2022, August 2). IsaMill Signature plot Procedure. Brisbane, QLD, Autralia.

Glencore Technology. (2024). *Malvern Procedure-Rev 1, Standard Operating Procedure.* Brisbane: Glencore Technology.

Gurnett, I., & Johnston, K. (2025). Specific Energy - The Race to the Bottom. *Comminution 2025.* Cape Town: MEI Conferences.

Kovalcik, A., Machovsky, M., & Wimmer, R. (2012, May). *Viscoelastic Properties of Mineral-Filled Poly(lactic acid) Composites*. Retrieved from Research Gate: https://www.researchgate.net/figure/SEM-micrographs-of-mineral-fillers-a-Mica-monoclinic-tendency-towards-pseudohexagonal_fig3_258382918

Larson, M., Anderson, G., Barns, K., & Villadolid, V. (2012). *IsaMill- 1:1 Direct Scaleup from Ultrafine to Coarse Grinding.* Brisbane: Glencore Technology.

Malvern. (2014). *Basic Principles of particle size analysis.* Retrieved from https://www.atascientific.com.au/wp-content/uploads/2017/02/AN020710-Basic-Principles-Particle-Size-Analysis.pdf

Malvern. (2014, August). Reliable Particle Sizing Mastersizer User Course. Sydney, NSW, Australia.

Russo, M., & Campostrini, I. (2008, January). Storia termica di una fumarola del Vesuvio (Campania, Italy) attraverso lo studio di minerali presenti su di un micro campione. Campania, Italy.