TECHNotes

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Error and Uncertainty in Metallographic Measurement

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Introduction

The idea of using 'uncertainty' has been slow to be adopted in many industries – this is perhaps understandable given the length of explanation required to cover all aspects of the general guide (134 pages in the GUM published by the Joint Committee for Guides in Metrology). However, the reason for its introduction is to pursue a better understanding of the causes of differences in measurement, as well as to promote the consideration of causes of uncertainty. The basic principles are quite straightforward, and when we apply them to daily practice in the workplace – as well as, perhaps, to our approach to development of Standards – we can be better positioned to understand and control the measurements that we make to reduce uncertainty and measure more precisely.

In most fields of scientific research and industrial quality assurance, we are constantly striving to improve the performance of the products and the materials from which they're made. This can sometimes be achieved through breakthrough technologies, but a significant proportion of progress is through incremental improvement, driven by enhanced understanding of the performance characteristics and properties of the material. This can lead both to the refinement of key properties and to reducing margins built into the design. For example, a soda can may be designed with a certain thickness because the material and structural characteristics are sufficient to prevent the can from rupturing in use. The thickness of material in the design will include a margin of safety – dependent on factors such as the variability of the material, the manufacturing process, the conditions of use, the acceptable failure rate etc. These factors along with unaccounted factors that were not considered during design, each will have their respective uncertainties. If we can control these various contributions to uncertainty, we can reduce the required margin built into the design. In this example, the benefit might be to reduce the amount of material used to make the can, and therefore the manufacturing cost. This can be achieved through material improvement and better control of manufacturing – but it can also be achieved by improving measurement confidence.

Mathematical analysis can quantify uncertainty, but does not always address how it may be improved. Understanding potential sources of error and, as importantly, their relative contribution, can be essential to addressing the need to improve both reliability and reproducibility of results.

The implementation of uncertainties into Standards has generally focused on providing a method to characterize equipment used, and the built-in process associated with it. Merging this concept with existing standards has some challenges, typically in translating the mathematical concepts to practical and usable methods. It's not the purpose of this paper to address these challenges, but instead to approach the subject from the other direction – how can the user look at components of uncertainty in their own methodologies and control them more effectively.

Definitions of Error and Uncertainty

It's helpful to start off with some definitions. From JCGM's Guide to Uncertainty Measurement [1]

"uncertainty (of measurement): parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand

measurand: A quantity intended to be measured"

When we stop to consider the measurand in any scenario, we may quickly realize that the definition of the measurand is important to improving certainty. Using the soda can example, if we wish to improve the measurement of thickness we could consider all the things that may affect the measurement. We would typically think of the measurement device as controlling accuracy, but other factors such as the location and number of measurements as well as alignment of the measurement device and environmental factors such as temperature may also affect the measured value. If we list and control those factors, we may reduce the uncertainty in our result. In this example, we would redefine the measurand from "measure the average thickness" to a more detailed instruction to control the spacing, temperature and alignment. This is, of course, exactly what we would wish to do in creating a Standard for measurement! By suitably controlling the definition of the measurand, and addressing sources of uncertainty, we improve accuracy, repeatability and reproducibility.

Measurements in metallography can present a more complex challenge. The standards aim to define the measurand, but the condition of the item being measured (the polished specimen), as well as the equipment used to do the measuring can be relatively uncontrolled and each factor can add to uncertainty. The objective of this work is to separate and examine some of the individual sources of uncertainty that are typical of measurements in optical examination of metallographic samples.

Experimental Method

To demonstrate the effect of varying a range of parameters on the microscope and imaging camera, a relatively simple sample was selected, of Carbon Fiber Reinforced Composite. This is a product consisting of layers of woven mats of carbon fiber encapsulated in resin. The properties of the material depend on the layup of the fiber, the quantity of fiber relative to the resin and the size and type of voids within the component. In the samples used, there is one readily recognizable harder phase (the fiber) surrounded by the softer polymer matrix. The effect of variables will be more readily recognized on a sample of this type.

To demonstrate effects on a more complex scenario, a WC-Co thermal spray coating has been used. This type of coating is typically applied to improve wear properties of a component. The coating is a moderately complex structure; the size and type of voids as well as the size and shape of features within the coating such as unmelted particles, oxides, interface features and cracks are all important to quality control.

Preparation Factors - Unrecovered damage

It's well understood that the process of metallographic preparation is aimed at removing damage from the specimen such that the true structure can be analyzed. However, it is not always a straightforward matter to recognize, or remove, remaining damage. This may be due to complex structures in the specimen, components that are particularly sensitive to damage from the preparation process, or from scenarios such as failure analysis where defects can be more complex or R&D where the structure and properties may be less well known. This is far too wide a subject for a short paper, but an example helps to illustrate the problem.

Figure 1 shows porosity measurements on a WC-Co spray coating at different stages of preparation . Even with the correct preparation stages being used, each stage of preparation must be done correctly and for long enough to recover pre-existing damage. In the example shown in Figure 1, the porosity was measured during each stage. The apparent porosity has been reduced by a factor of three in just the last few stages. In this case, with knowledge of the material, experimentation with different preparation options and observation of detail in the coatings, we would conclude that the final measurement was accurate.

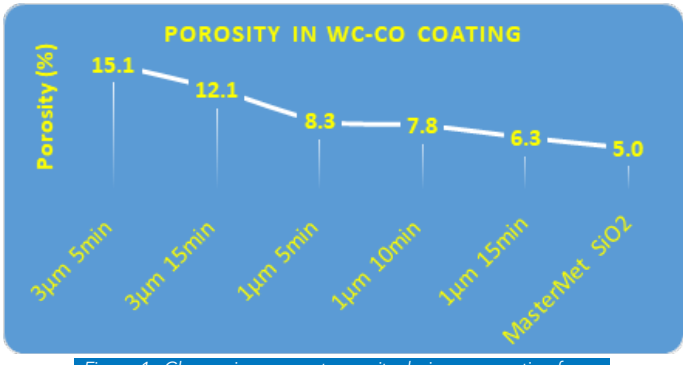

Figure 1: Change in apparent porosity during preparation for a WC-Co spray coating

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ASTM URL: http://www.astm.org, DOI:10.1520/STP160720170221

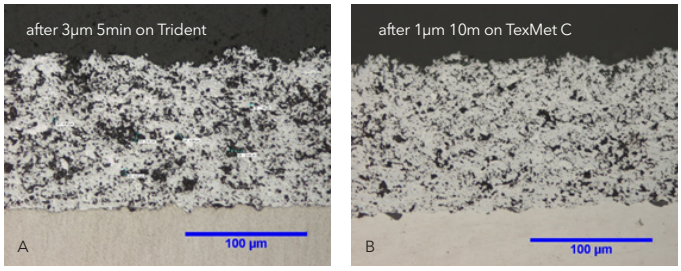

Figure 2: WC-Co samples during diamond preparation stages (a) has 15% porosity (b) has 8% porosity

Clearly the level of unremoved damage in this sample has a significant effect on measurement result. The correct metallographic recipe depends on the material being prepared, but typically a good understanding of the characteristics of the material being examined along with appropriate inspection can help us identify good preparation. Prior to the thermal spray process, the surface of the substrate material is roughened by grit-blasting to promote mechanical adhesion. Inspection of particles remaining at the interface and clarity of features and edges are just some of the things we can use to give an indication of effective preparation.

Figure 3 (a) shows a poorly prepared specimen with significant remaining damage – the grit particle is fractured; the boundary is ur… me… , a…

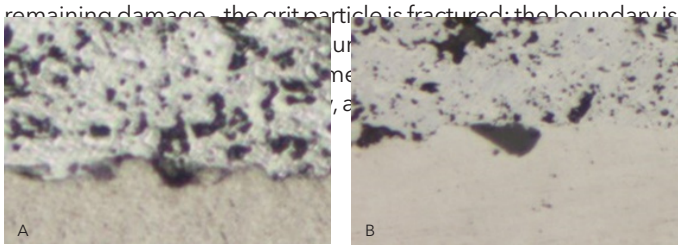

Figure 3: Same specimen as Figure 1, digitally magnified to show small detail. (a) shows partially recovered damage, grit particle is broken and there is edge damage from grinding step at the interface and (b) showing complete grit particle with no visible damage at interface.

Preparation Factors – Polishing relief

A common mistake in metallography is to extend polishing times (most often on soft cloths) to recover visible damage remaining at the end of a preparation route. Extended polishing on soft surfaces can lead to polishing relief – areas of the same specimen polish at different rates, and a height difference is generated. As light microscopy relies on reflection to produce contrast in the image, the slope between areas with polishing relief will appear darker, or even as a black line. For unsupported areas and edges, this can prevent accurate measurement. In the example below, an over-estimate of porosity size is likely due to the rounding at edges

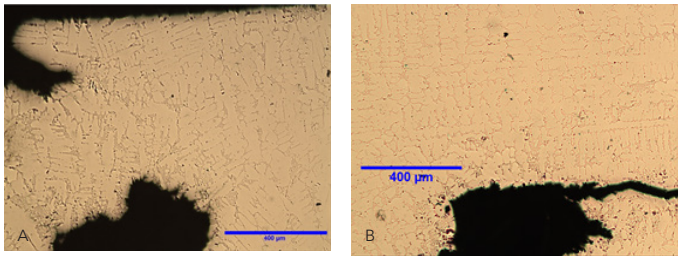

Figure 4 – the effect of (a) over-polishing versus (b) good preparation on apparent porosity in a nickel superalloy

It's therefore good practice to minimize polishing time on soft surfaces, and the correct action would be to ensure that the penultimate stage:

(i) keeps the specimen flat, typically by using a harder/flatter preparation surface and

(ii) allows for a short polishing step – the difference in abrasive size should not be excessive, so that damage such as scratches can be removed in a relatively short time

Microscope and Camera Settings

By using a field of view on a single sample and varying one parameter at a time, we're able to isolate the effect of that parameter. This was done for a variety of camera settings, and the percentage change in apparent fiber content for a CFRP sample was measured, using grayscale based thresholding with visual confirmation of upper and lower threshold values. All tests were done with a single operator and all conditions that were not being changed were held constant, where possible. A Nikon MA200 inverted compound microscope was used, along with a CCD digital microscopy camera with a ½" chip. Omnimet digital imaging software was used for capture, digital transformations, and image analysis. A mixture of microscope and camera settings were modified, as these are often intrinsically linked in today's metallography laboratory. To maintain best comparability between images, the overall brightness was controlled in every case, such that the maximum pixel brightness was just below saturation.

The graph in Figure 5 shows the overall results of each of the factors investigated, expressed as absolute value % variance from a baseline measurement. The largest variations (outside the 95% confidence interval from the tests for baseline value) are marked in orange.

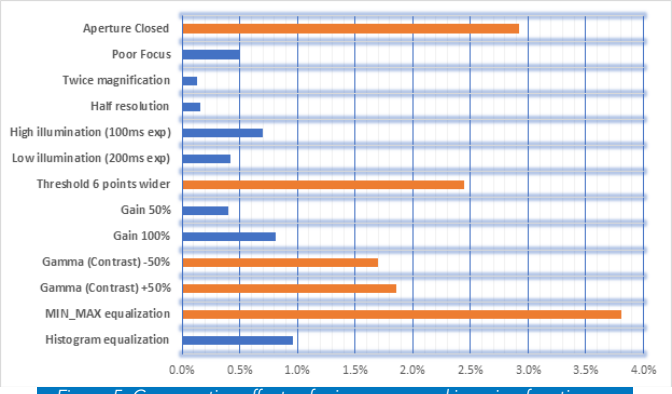

Figure 5: Comparative effects of microscope and imaging functions (% change in measured porosity)

Some of the factors that typically would draw more attention – such as camera resolution, and poor focus – had a relatively small effect. At higher magnifications, resolution is often limited more by the optics than the camera. At lower optical magnification, camera resolution becomes more significant. If the feature size of the structure being observed were smaller, we may expect these parameters to have a much more prominent effect. We similarly found that illumination, exposure time, and the use of 'gain' had relatively small effects on our measurement, if the overall distribution of brightness in the captured image did not get to saturation.

Looking more closely at the remaining high-impact criteria can help us to improve measurement certainty the most.

Microscope Factors- Aperture Diaphragm

Almost all compound microscopes have an aperture diaphragm. This device modifies the size of an aperture in the light path of the microscope, and modifies the amount of light passing through as well as affecting focal depth, resolution and contrast. Many operators are not aware of the correct use of this function, but it can have a significant effect on measurement. When resolution is reduced, darker areas appear to 'grow' and thresholding (defining the boundary between light and dark) becomes more difficult. Figure 6 shows the same field of view on a CFRP specimen with the aperture fully open (a) and closed (b).

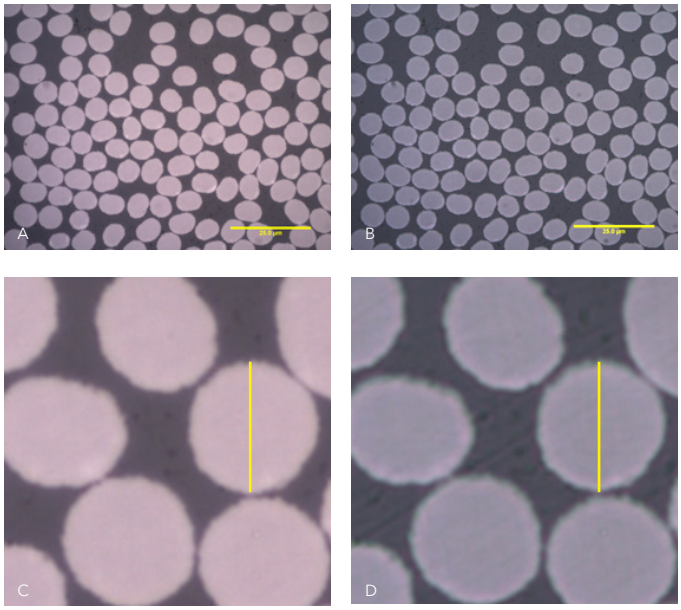

Figure 6: CFRP specimen at 50x objective magnification: (a) aperture open; (b) aperture closed; (c) digitally magnified area from (a); (d) digitally magnified area from (b)

Digitally enhancing these images shows some visible difference in contrast and resolution – fine scratches from 0.3µm alumina can be seen in the polymer matrix in (d). However, when we take a measurement from these images the linear distance shown in (d) is 4% shorter than the line shown in (c) – even though the measurement itself appears the same. This is attributable to the difference in resolution from the change in aperture setting. The recommendation for best practice is thankfully straightforward. The aperture diaphragm should generally be open when making measurements to maximize optical resolution. Other important criteria to optimize resolution are centering and set-up of the lighting and a good maintenance/cleaning schedule.

Image processing factors - Thresholding

Thresholding is a common process by which we identify different areas of an image, to enable digitized measurements. The principle is straightforward – a typical grayscale image has 256 gray levels. If we plot out a diagram of gray level against number of pixels we can identify peaks. When we use software to help us identify a phase under the microscope, we are setting 'threshold' points to characterize the areas of the image. In the example shown in Figure 7, two regions can be clearly defined – the region from 0 up to line A and the region from line C to maximum. The region between A and C appears fuzzy and it may not be easy to define exactly where a single line separating the two peaks should be placed.

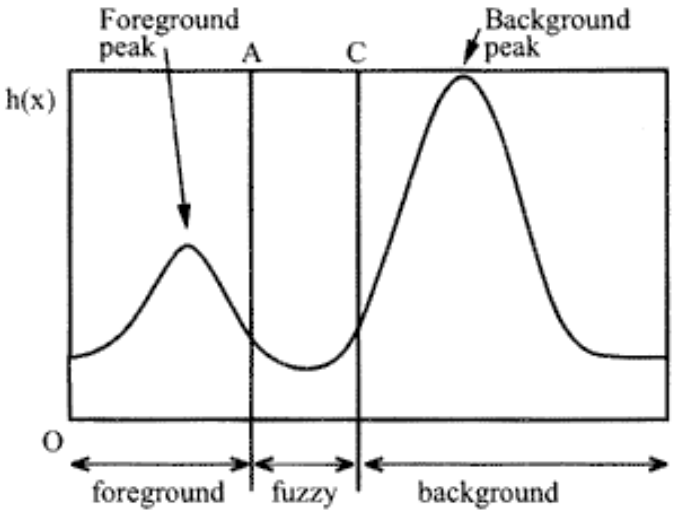

Figure 7 Representative image of intensity histogram, from Devi [3]

When we have poor resolution, or edge rounding, the 'fuzzy' area tends to be larger, and the minimum point may be skewed, due to the uneven effects of intensity distribution. This can therefore give us more variability in results (greater uncertainty), as well as a shift in mean measured value (error).

Image processing algorithms, and often automatic thresholding tools, can vary greatly in their success in finding this threshold point. In the CFRP example used, the 'fuzzy' area was quite small, as the material was well prepared with minimal edge rounding and good contrast between phases. This helps to keep the thresholding more reproducible, and automated systems are more likely to work effectively. It's also strongly recommended to maintain a consistent image brightness between specimens.

When the 'fuzzy' area is large, it's tempting to use image processing to improve the contrast of the image. Image processing is not new, and has been successfully used in many applications for decades. However, as technology has advanced the use of image manipulation algorithms has become familiar, for instance in photo editing software freely available with just about every 'smart' device. There's a perception that image 'enhancement' is beneficial – and indeed in many applications this is the case. However, when we're taking precise measurements from images it's important to appreciate that some of these functions may change our results.

Image processing factors - Image Contrast

Gamma adjustment is a tool available in many image processing applications. Essentially, it modifies the distribution of intensity in the histogram. Our eyes perceive image contrast more effectively at low levels than at high, and so when we look at modified images, such as the below, we tend to 'prefer' the look of image (b). In fact, this modification changes the distribution function and therefore the apparent thresholding point, and our measurement changes.

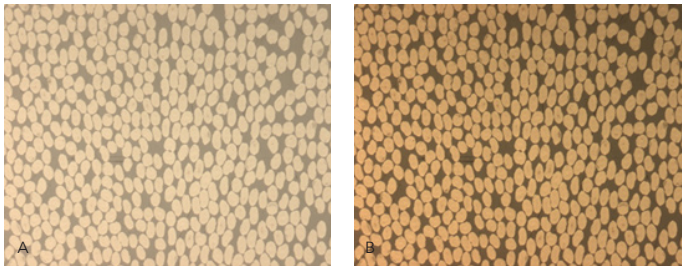

Figure 8: CFRP surface (a) as-captured and (b) after enhancing with Gamma contrast adjustment

Histogram equalization is another method of modifying contrast in an image. In this case, the intent is to take an image with a poor range of contrast levels and expand it across the available range. In our example, the effect of this process was not severe – but it has just as much potential to modify measurements as the gamma adjustment. This principle is true for many types of image enhancement, and it's recommended to avoid these whenever possible.

Conclusions

Most of the principles discussed in this paper are relatively straightforward, and it should be a simple matter to understand and control in the laboratory. There are some key recommendations that will help minimize error and uncertainty in optical analysis of metallographic specimens.

- Keep specimens flat throughout preparation and minimize polishing relief.
- Ensure the microscope has high quality lighting (Koehler illumination) and lenses (particularly numerical aperture rating)
- Optimize resolution of the microscope using good maintenance and adjusting correctly.
- Avoid changing the captured image. Create contrast with the specimen and microscope, and not through digital enhancement.
- Recognize and account for Uncertainty where possible. Understand the tools and processes being used to avoid un-necessary error.

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