In-situ mechanical performance testing of materials using SEM and TMA: Examples, comparisons and sample preparation

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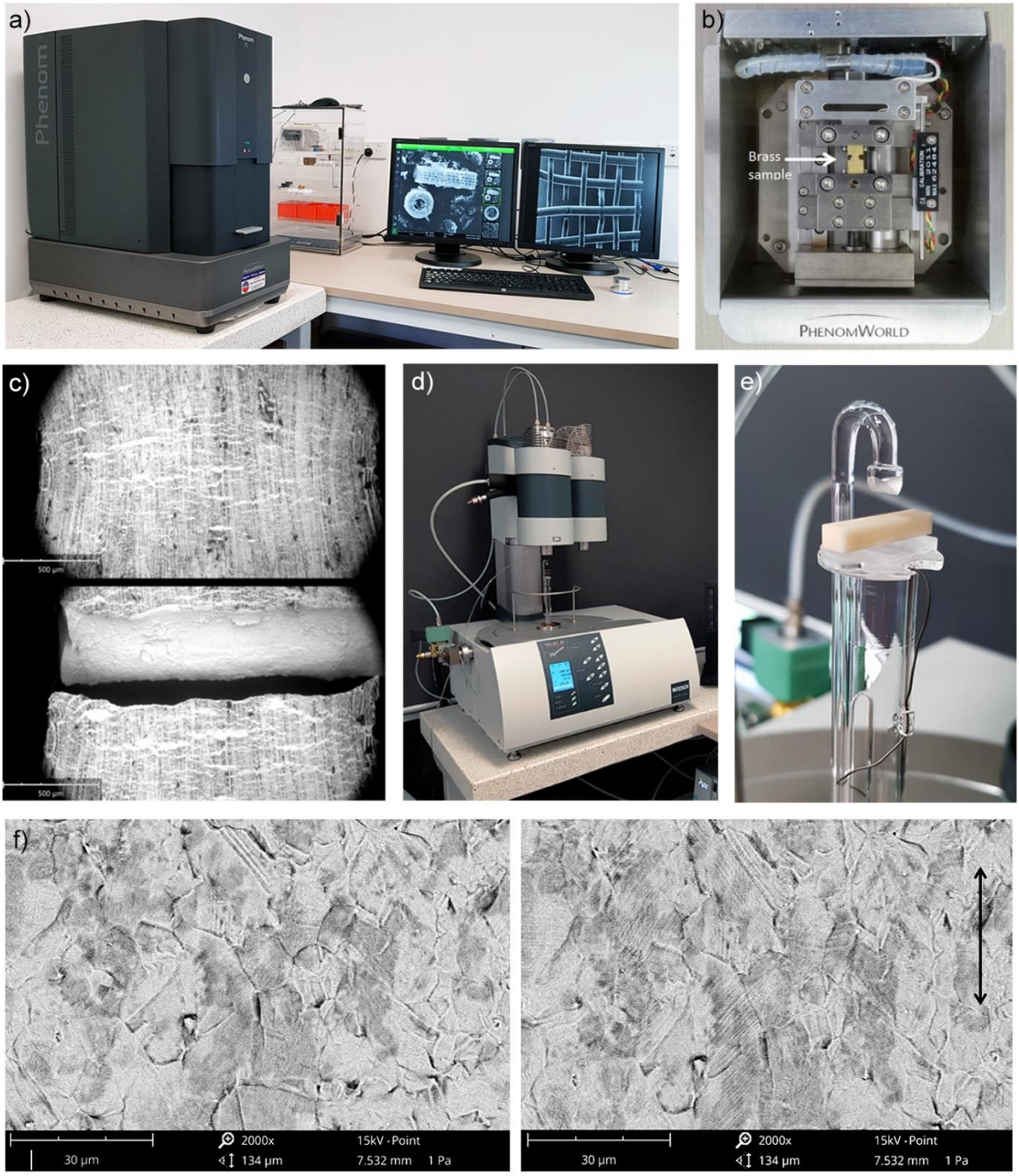
Characterisation, mechanical performance and failure analysis of both traditional and new composite materials are important parameters required for engineering and material science applications. Users often need to determine the effect of mechanical properties on materials under different temperature environments, and find the location where a material has failed.

Real time studies of materials being mechanically tested, the location of failure and most importantly the mode of failure, give a more precise understanding of a material's performance. While Scanning Electron Microscopy (SEM) can be used to find the location of failure in materials, here a Phenom XL Desktop SEM with an integrated tensile stage allows *in-situ* mechanical testing with loads up to 1000N. This means the user can observe failure as it occurs while acquiring data on its mechanical properties such as tensile strength and Young's modulus. Short videos can also be made using this setup. Results were then compared to those obtained from mechanical testing in a Netzsch Thermo-Mechanical Analyser (TMA), which can be used to determine the effect of different temperatures on sample mechanical properties.

For these experiments, care and forward thinking are needed. Current research has shown that with proper sample preparation, better visual failure investigation of a material is possible. For example, brass dumb-bell shaped sections can be carefully polished and etched on the surface prior to *in-situ* mechanical testing. This preparation of the surface allows greater surface detail visualisation of the failure mechanisms around grain boundary regions and also aids in a better understanding of failure modes.

This presentation will compare results from a number of different materials investigated in an effort to obtain a better understanding of material performance and failure modes. It will also touch on best practice techniques for sample preparation. Both *in-situ* SEM and TMA experiments are not always straight forward to run, and can be quite challenging to obtain good clear results. There are also a number of factors that need to be taken into account, particularly when setting up an experiment, such as gripping the samples, keeping samples focused and predicting where failures will occur. Further to this, subsequent backscattered/secondary electron imaging (BSE/SEI) and EDS microanalysis of the failed sections allows for further information on failure sites, such as analysis to find any chemical impurities.



Figures a – f : a) The Phenom XL SEM located at the AMCF, b) the *In-situ* SEM tension stage with a brass sample sitting in the centre, c) *In-situ* SEM images of an unpolished brass sample before and after fracture, d) the Netzsch TMA setup, e) TMA setup for a three point bend test and f) *In-situ* SEM images of an etched brass sample before tension (left) and under tension (right). Here the grain boundaries can be seen. As the sample is put under tension, stretching of the grains can be seen as well as the appearance of micro-fractures (black arrow shows direction of tension).