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Overcoming Challenges in Material Science Testing with the use of Large Specimen SEM Analysis

Adriana Romero
VisiTec of America LLC
Knoxville, TN
romero@visitec-em.de

One of the most oft-confronted challenges in materials science, when using scanning electron microscopy, is the limitation of the size of the sample being examined, as well as the analysis application planned. Normally, only small samples in the order of ten to a hundred millimetres in diameter may be investigated. In addition, until now, conventional high-load fatigue testing inside a scanning electron microscope (SEM) seemed impossible. Nevertheless, SEM testing is a very valuable method in the fields of material and biological science as well as in quality control and failure analysis. For many technological interesting applications, conventional scanning electron microscopy is not possible because the specimens that need to be investigated cannot be destroyed for the SEM investigations for one of several reasons. It may be that the specimen must be kept whole so that it can be returned to a production line or perhaps the specimen is an object of historic and/or artistic interest and value.

Fig. 1

Positioning system axis drawing

Focusing on Scanning Electron Microscopy

Although SEM testing is a well established technique in industry and research, many of the technical features, in particular the maximum resolution, of a standard SEM have been increasing enormously during the last decade, mainly because of the introduction of the Field Emission gun and the increase of the quality of the lenses. In addition to the advancements in electron optics, analytical systems like Energy Dispersive Spectrometry (EDS) and Electron Backscattered Diffraction (EBSD), and their software systems, have improved their quality and ability to perform and deliver better analysis and give better quality results. These systems have been improved through engineering to have much better handling and control of the complete system.

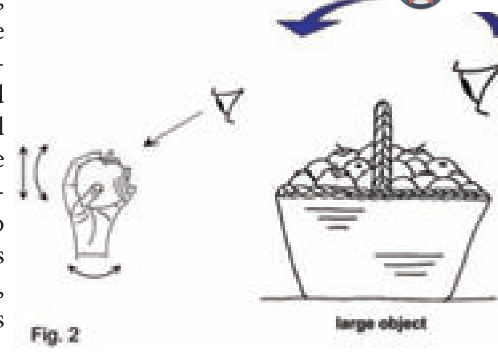

Fig. 2

Human behavior drawing

The first step in overcoming the challenge of specimen size limitation, was taken by VisiTec Microtechnik in Germany, when in 1994, the Large Chamber SEM was introduced and accepted within the scientific community. The large chamber, which can accommodate samples up to 1000mm in diameter, is equipped with an innovative positioning system that allows views from different angles by moving the electron gun and detectors, as well as the sample (fig.1) identical to the behaviour of human beings. If humans look at a small object, they will turn the object around, with the human eye fixed. When looking at a larger object, the human moves around the fixed object in order to observe it completely (fig. 2). The first case is similar to the situation in standard SEM's, while the latter reflects the situation for the LC-SEM.

In addition to the investigation of large samples, the LC-SEM also has a great potential for *in-situ* observations of the deformation behaviour of materials, as well as for relatively small production processes in the field of micro-system techniques. The Large Chamber SEM also makes it possible to perform "interrupted *in-situ*" experiments for larger engineering parts. As an example, testing the tribological behavior of highly loaded parts from high-pressure

Fig 3

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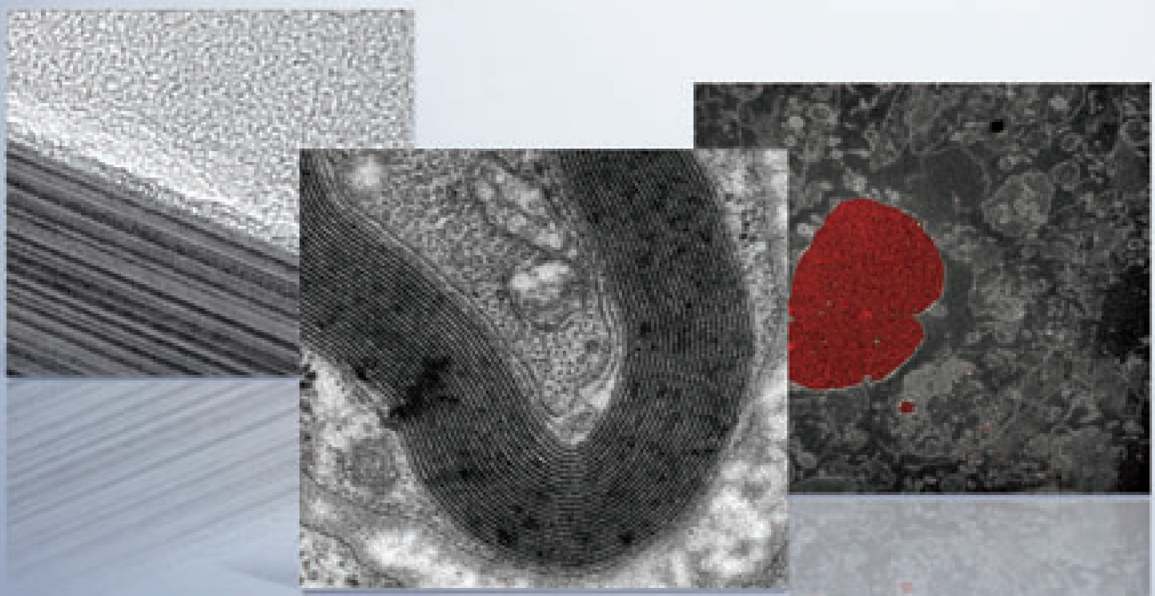

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pumps can be investigated by interrupted monitoring. The parts can be in service for a period of time, followed by an investigation of the loaded surfaces in the LC-SEM, returning them to service immediately after the investigation is completed. This way of monitoring a system will open a broad field for engineering applications that will allow the investigator to get a closer and a more detailed understanding of the running-in and damaging processes.

A breakthrough application

To meet the need of performing *in-situ* investigations of deformation behaviour, a new design of the LC-SEM was created, where a servo-hydraulic fatigue-testing frame from MTS was integrated into the chamber. Thus creating new possibilities for materials testing and overcoming the challenge of performing large specimen fatigue testing in an SEM. The idea of combining these two large components seemed impossible. Just the integration of the hydraulic components inside a vacuum environment was the beginning of many design related challenges to overcome. In fact, the integration of detectors and analytical equipment in a vacuum environment represent a challenge for the design and precise operation of the system. Integrating an EDS system brings many benefits to complete the analysis. Incorporating an EBSD system allows the investigator to have the ability to determine relevant crystallographic analysis of engineered parts, which can have a strong input for solving technological problems as well as for quality control.

The fatigue-testing equipment is mounted on a steel frame and introduced inside the vacuum chamber. For this process there is an opening left in the chamber, that will allow the use of the fatigue testing equipment by itself not under vacuum and subjected to SEM analysis, or the opening can be closed with a flange to create the vacuum and use the two devices at the same time (fig.3). In this combined test, not only the force and break point of the material is determined but also the crack is available to be viewed and studied thanks to the microscope. By these means, the microscope transforms itself into a complete testing device; since the properties of the materials crystal grains can be studied *in-situ* after the fatigue testing is performed, delivering complete test results. This system increases the researcher's ability to record material structural changes that precede crack nucleation and allows observation of the influences of microstructure on the early stages of crack propagation. The design of the system includes a node control mechanism that will create a still observation node at any location on the specimen as the fatigue cycling occurs, allowing a point of interest to remain within view. The exceptional stability of this machine enables improved *in-situ* study of the fatigue-cracking phenomenon.

The combination of the LC-SEM and the Servo-hydraulic Fatigue Testing Machine facilitates mechanical tests on standard specimen geometries. Moreover, under cyclic loading, all the classic control channels–stroke, stress and strain–of a standard fatigue-testing machine are available. For the first time, a real closed looped controlling and cyclic deformation test under plastic strain control can be realized for an *in-situ* test rig. Since many tests require high temperatures in order to be performed, it is also possible to heat the specimen up to 1100°C, which when combined with the EBSD integrated in the chamber, makes it possible to obtain relevant crystallographic data with respect to the applied deformation at temperature. Furthermore, this complete system will allow, for the first time, *in-situ* observations in the SEM of metal forming processes that might provide valuable data not only in the field of micro-system forming processes, but also for many other technologically interesting sheet-forming processes. (fig. 4)

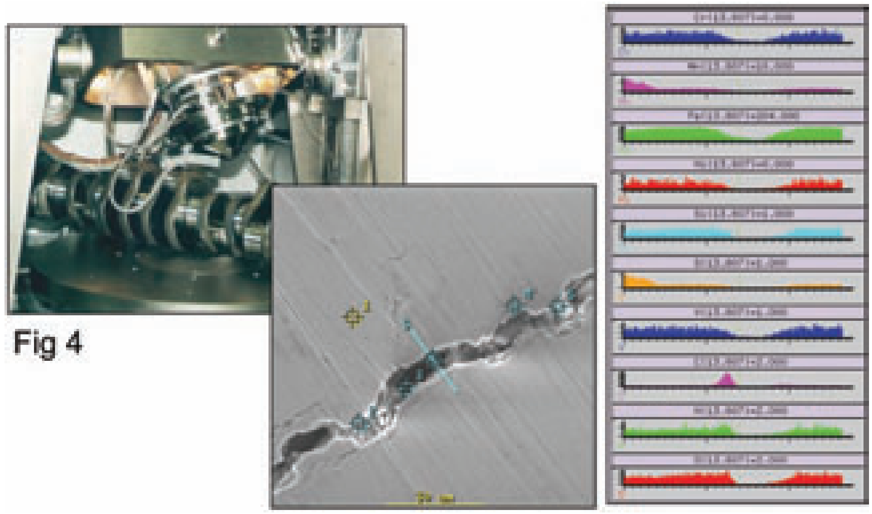

Fig 4

EDS Analysis and Crankshaft

More generally, this system provides a great potential to gain more insight on relevant deformation mechanisms for many interesting problems. One example is the investigation of the mechanisms responsible for the so-called "Paradox of Ductility and Strength," as it is observed in ultra-fine grained materials. Basic deformation or metal forming processes within the context of shifting limitations of currently used materials or technological processes may be studied.

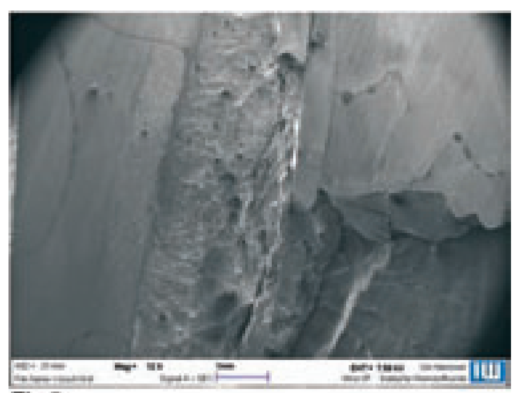

Fig 5

SEM of defect in surface of Crankshaft seen in fig. 4.

The LC-SEM with the integrated fatigue-testing machine developed by VisiTec Microtechnik GmbH and installed in University of Erlangen-Nürnberg in Germany, is perfect for any application where stress of a specific material needs to be measured and also the study of the crack and crystal grain behavior properties are important. In addition, the main advantage of the integration with the LC-SEM is that the standard part can be tested, instead of using a small sample. In this way, a sample can be fully examined with only one instrument, without cutting the sample and introducing new anomalies to the microstructure. ■

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