



**Material and failure analysis**  
**Root-cause investigations**

**Specification of services**

## Scope of work

As the basis for an assignment, IABG prepares an examination plan based on the information provided by the customer and with approval of the customer in accordance with the described service package(s). The examination plan equals a fixed price offer. Orders must be in writing. The final schedule will be agreed on after receipt of the order.

A minimum order quantity of 500,- Euro applies to the above services. In case of urgent orders, i.e. execution within 24 hours, an additional charge of 50% is applied.

### 1 Sample preparation

#### 1.1 Preparative work (machining and factory)

Transportation (incl. usage of on-site cranes and forklifts) and disassembling of the test items on the IABG premises in Ottobrunn, use and provision of machine tools and separating tools, cutting of modules / test items including skilled staff.

#### 1.2 Preparative work (laboratory)

Use and provision of laboratory equipment and resources. This applies to cutting services for sampling, installation of laboratory experiments, customer and project tailored services.

### 2 Macroscopic documentation

#### 2.1 Macro documentation

Macroscopic analysis of test items, macro-documentation with macro lens and digital camera, stereo microscopic fracture analysis, integration of the pictures in our Image Access database.

### 3 Metallographic Investigation

#### 3.1 Metallographic specimen including microstructure examination

Micro- or macrosection preparation of a sample extracted at standard contrast within a root cause analysis. A light microscopic microstructural examination to record microscopic features such as microstructure, structure, defects, coatings, segregations, etc. Including the determination of microhardness of the core structure with five individual measurements and the respective mean value.

- ⇒ Maximum sample sizes (embedded) Ø 50 mm / 35 x 65 mm
- ⇒ 50% surcharge for urgent examinations with completion within 48 hours
- ⇒ Special sizes on request

#### 3.2 Metallographic specimen incl. basic documentation

Micro- or macrosection preparation of a sample extracted at standard contrast. Documentation of the microstructure including five images will be provided.

- ⇒ Maximum size of the (imbedded) test items Ø 50mm / 35 x 65 mm
- ⇒ 50% surcharge for urgent examinations with completion within 48 hours
- ⇒ Special sizes on request

#### 3.3 Metallographic specimen preparation / higher number of samples

Embedding of sample including metallographic preparation at standard contrast. Ideal for large quantities of samples with identical geometries. Contains basic documentation of optical microscopy.

#### 3.4 Light microscopic investigation

Multi material application, light microscopic examination of microstructures on prepared macro- and micro-sections.

### 3.5 Standard analysis materialography

Accredited tests including test certificate for the structure analysis below:

- ⇒ Grain size according to DIN ISO 643, ASTM E112
- ⇒ Coat thickness of metal and oxide coats, microscopic procedure according to DIN EN ISO 1463
- ⇒ Examination of non-metallic, carbidically and sulfidic inclusions according to DIN 50 602 (Procedure M), ASTM E 45, SEP 1520, SEP 1615
- ⇒ Microstructure of cast iron according to DIN EN ISO 945 (analysis surface up to 80 mm<sup>2</sup>)
- ⇒ Porosity analysis according to VDG recommendation P201E, VW5009, PV6097 (test surface up to 80 mm<sup>2</sup>)
- ⇒ Particle analysis (statistical analysis / classification of maximum 3 light microscopy or REM-pictures)

## 4 Scanning electron microscopic investigation

### 4.1 Sample vaporization with gold

Vaporization with gold (Au) per REM-specimens for non-conducting materials as plastics, biological specimens and ceramics.

### 4.2 Scanning electron microscope investigation including EDX

Provision and use of the scanning electron microscope (REM) with an energy dispersive x-ray microanalysis (EDX). For investigations, tungsten and field emission devices are available. Both conductive (metals) and non-conductive (plastics, ceramics, biological) materials can be examined.

- ⇒ Fractography – analysis of the fracture surface for determination of the failure mechanism
- ⇒ High-resolution topography analyzes at a maximum resolution of 0.6 nm
- ⇒ Qualitative and semi-quantitatively microanalysis (EDX)
- ⇒ Point, integral, line and object analysis, spatially resolved microanalysis (mapping)
- ⇒ Customized analysis/examinations

## 5 Mechanical technological analysis

### 5.1 Macrohardness (HB, HR, HV, HL)

Accredited test on calibrated hardness tester including test report (5 single measurements, averaging) for each of the following test methods:

- ⇒ Brinell (DIN EN ISO 6506-1, ASTM E10)
- ⇒ Rockwell (DIN EN ISO 6508-1, ASTM E18b)
- ⇒ Vickers (DIN EN ISO 6507-1, ASTM E92)
- ⇒ Mobile Hardness test according to Leeb (HL-C, -D, -G, DIN50156-1)

### 5.2 Low load and microhardness (HV1 down to HV 0.01)

Accredited test according to Vickers (DIN EN ISO 6507-1, ASTM E92) on calibrated hardness tester including test report (five single measurements, averaging).

### 5.3 Hardness profile (DS, CHD (Eht), Nht)

Accredited test according to Vickers (HV1 down to HV 0.01, DIN EN ISO 6507-1, ASTM E92) on calibrated hardness tester including test report and graphical illustration of the hardness profile. Evaluation of the hardness profile with a maximum of 20 single measurements for each of the following tests:

- ⇒ Case hardening depth according to DIN 50 190-2, DIN EN ISO 2639 and DIN EN 10328
- ⇒ Decarburization depth according to DIN EN ISO 3887

- ⇒ Nitriding hardness depth according to DIN 50190-3
- ⇒ Melting hardening depth and melting depth according to DIN 50190-4

#### **5.4 Hardness profile (welded joints)**

Accredited weld seam hardness test according to DIN EN 1043-1 or DIN EN 1043-2 on calibrated hardness tester including test protocol.

#### **5.5 Hardness test on plastics and rubber**

Accredited hardness test in accordance to Shore A and D (DIN EN ISO 868) and IRHD (DIN EN 48) on plastic and rubber material including test protocol.

#### **5.6 Tensile test - sample manufacturing**

Manufacturing of cylindrical short proportional test specimen. Samples are prepared (depending on the available resources) directly at IABG or with eligible subcontractors of IABG.

#### **5.7 Tensile test – testing**

Implementation of an accredited tensile test on calibrated testing machines with precision strain measurement and determination of characteristic values ( $R_p$ ,  $R_m$  and  $A_5$ ) including test report.

The tests are performed (depending on the available resources) directly at IABG or with a certified and accredited subcontractor of IABG.

#### **5.8 Charpy impact test – sample manufacturing**

Preparation of three samples (V-notch) according to ISO 148/EN10045-1. A qualified subcontractor of IABG will perform the preparation.

#### **5.9 Charpy impact test – testing**

Implementation of the impact test and determination of the impact energy including test report of three samples. The tests are performed (depending on the available resources) directly at IABG or with a certified and accredited subcontractor of IABG.

### **6 Analytics**

#### **6.1 Chemical material analysis (spark emission spectroscopy)**

Quantitative emission spectroscopic analysis by a certified and accredited subcontractor of the IABG including a test report.

#### **6.2 Remelting for chemical material analysis**

Remelting of small samples into compact analytical samples (Fe, Cu, Al-base) or cast iron samples into a white solidified state. A certified and accredited subcontractor of the IABG provides this service.

#### **6.3 Residual stress**

X-ray determination of residual stress at the material surface by a qualified subcontractor of IABG. Includes two directional measurements (0 ° and 90 °) per measuring point.

#### **6.4 Thermogravimetric analysis (TGA)**

Determination of humidity, fiber and resin content in plastics and fiber-reinforced plastics using a thermogravimetric measuring system with automatic measuring cycles. Multiple analyses with high initial weights up to 5 grams and max. 1,000°C possible.

Fiber volume is obtained by determination of density (buoyancy-flotation method) according to DIN EN ISO 1183-1 (procedure A) and by application of a macro TGA (thermogravimetric analysis). For the determination of the fiber and resin volume content, the densities of fiber

and resin are required. Provision of only the respective mass share in case the densities are unavailable.

### **6.5 Dynamic differential calorimetry (DSC)**

Using the dynamic differential thermoanalysis the glass transition temperature and the reaction enthalpy of plastics according to the specifications ISO 11357-2/-3 is determined. Temperature within the -90°C to +450°C range can be chosen. Identification of heating curve for each sample at standard heating rate of 10 K/min.

For the determination of curing effects on reactive resins, the reaction enthalpy ( $\Delta H_{100}$ ) of the fresh material and the resin mass share of the cured specimen are required. Alternatively, the enthalpy values are determined after post-curing.

A certified and accredited subcontractor of the IABG provides these tests.

### **6.6 Dynamic mechanical analysis (DMA)**

Performance of dynamic mechanical analysis of fiber reinforced plastics (FRP) in accordance to DIN 65583 as single bending fatigue test (single cantilever). Determination of glass transition temperature by the temperature dependency of the elasticity module. Evaluation of storage and loss modules.

Standard tests include a heating rate of 5 K/min, a frequency of 1 Hz, an amplitude of 15 microns, a temperature range within + 40°C to + 270°C. The minimum sample geometry is 35 mm x 10 mm x 2 mm. Drying of specimen at 105°C commences 7 days prior to measurements.

A certified and accredited subcontractor of the IABG provides these tests.

### **6.7 Instrument-based analytics**

#### **6.7.1 Carrier Gas Hot Extraction (TGHE)**

Quantitative determination of hydrogen (H), nitrogen (N), oxygen (O), carbon (C), and / or sulfur (S) by a certified and accredited IABG subcontractor.

#### **6.7.2 X-ray fluorescence spectroscopy (RFA)**

Quantitative determination of elements with an atomic number greater than sodium by a certified and accredited subcontractor of IABG.

#### **6.7.3 Glow discharge spectroscopy (GDOES)**

Qualitative and quantitative determination of the elementary composition by creating a depth profile from the surface by a certified and accredited subcontractor of IABG.

#### **6.7.4 X-ray diffractometry (XRD)**

Qualitative (optional: semi-quantitative) determination of crystalline phases by a certified and accredited subcontractor of IABG.

#### **6.7.5 Fourier Transform Infrared Spectrometry (FTIR)**

Qualitative determination of organic compounds by a certified and accredited subcontractor of IABG.

#### **6.7.6 Gas chromatography with mass spectrometry coupling (GC / MS)**

Qualitative and quantitative determination of organic compounds by a certified and accredited subcontractor of IABG.

### **6.7.7 Time of Flight Secondary Ion Mass Spectrometry (ToF-SIMS)**

Qualitative (optional: semi-quantitative and spatially resolved) determination of the atomic and molecular composition in the uppermost 1 to 3 monolayers of a solid by a certified and accredited subcontractor of IABG.

## **7 Non-Destructive tests**

### **7.1 Visual test – VT**

Visual tests for the identification of macroscopic visible damages or deviations. Provision of inspection documentation including test protocol and possible findings.

### **7.2 Dye penetrant testing – PT**

Dye penetrant tests to detect defects open towards surface conducted by qualified and approved test staff according to DIN EN 4179 or DIN EN ISO 9712. Provision of inspection documentation including test protocol and possible findings. Testing is performed within the IABG laboratory or on-site.

### **7.3 X-ray inspection – 2 dimensional**

For the detection and measuring of inner structures and defects, NDT tests and failure analyses are performed using the 2D mode of a 180 kV microfocus x-ray system with a dynamic positioning of samples in X, Y, Z and R direction in relation to the x-ray. With the enlargement of the detector/sample distance, a magnification of structures up to 2000:1 is realizable. Customer attendance during such investigations is possible. Provision of inspection documentation (images of the investigated areas).

### **7.4 X-ray tomography 180 kV microfocus**

Scanning of previously prepared specimen at a horizontal 360° rotation, using operator agreed beam parameters in a digital X-ray system. Combination of single-slice images into a volume model using appropriate algorithms. Depending on the sample size, a maximum resolution of 3 microns is realizable. The detail recognition is at 1 micron.

### **7.5 X-ray tomography 180 kV nanofocus and 300 kV microfocus**

Examination in a digital X-ray system either with a 180 kV nanofocus tube or with a 300 kV microfocus tube (depending on the sample size, material density and required resolution) in accordance with WP 4.2. The metrology package enables precise measurement of the outer and inner geometries of the test specimen. The 180 kV nanofocus tube, allows a detail detectability of 200 nm. The 300 kV microfocus tube allows a detail detectability of 1 micron.

### **7.6 X-ray testing $\mu$ CT – data analysis and evaluation**

Analysis and evaluation of defects, structures and inhomogeneity detected in terms of shape, size and location in three dimensions. Length, area, volume can be measured. Furthermore, the nominal/actual value comparison of outer and inner geometries is possible. CAD-geometry data gained during the volume scan (in STL-data format) is exportable for CAD and simulation tools/applications. The evaluation is performed by usage of Volume Graphics Software System (VGL).

- ⇒ Provision of generated images, videos and measurements as well as the VGL viewer software to the customer.
- ⇒ Usage of the results from work packages 7.4 or 7.5 as a database for volume analyses and evaluations.



## **8 Powder Genetics**

### **8.1 Macroscopic features**

Visual and stereomicroscopical examination of sample powder at up to 40x magnification. Focusing on manufacturing related imperfections by detecting any color changes or contaminations. Furthermore, the investigation provides information on any existing agglomerations and the bulk behavior as a first indication of the respective moisture content and particle structures. For this examination, a minimum sample powder quantity of 5 grams is required.

### **8.2 Chemical powder composition**

Chemical analysis of metallic powders by means of X-ray fluorescence analysis. Preparation of RFA borate melts. Determination of the content of alloy and trace elements by means of element-specific fluorescence radiation. For this examination, a minimum sample powder quantity of 10 grams is required.

### **8.3 Determination of elements C, S, H, N and O**

Determination of the proportional of the elements carbon, sulfur, nitrogen and oxygen in metallic material by carrier gas heat extraction (CGHE). During the heating process of samples in an impulse oven, infrared and thermal conductivity cells measure the diffusing gas content. The detection limit is 20µg / g for the elements C, S, N and O. For this examination, a minimum sample powder quantity of 10 grams is required.

### **8.4 Determination of the water content**

Determination of the powders water content by Karl Fischer titration. Oven heating the sample commences at 200°C, transferring the released water into a reaction vessel. Coulometric titration of the water against iodine and measured electrometrically. For this examination, a minimum sample powder quantity of 10 grams is required.

### **8.5 Particle sizes, distribution, HDP, cavities, geometry**

A 3-dimensional model of individual sample powder particles is recorded using computer tomography. Determination of respective particle sizes and form factors including powder-grain-size distribution. Detection of further cavities in powder particles, degenerative particle structures and foreign material in the form of higher density particles (HDP). For this examination, a minimum sample powder quantity of five grams is required.

### **8.6 Topography and morphology**

Determination of Powder morphology in terms of sphericity, particle agglomerates, adherent nanoparticles, discoloration, and contamination. The powder examination is carried out using a scanning electron microscope. For this examination, a minimum sample powder quantity of 5 grams is required.

### **8.7 Analysis of Powder Particle Contamination**

Possible contamination of metal powder material will first be separated and afterwards analyzed in order to determine the type and content of impurities and foreign materials.

The respective examination steps depend on the type of powder material and the type of contamination. The performance of a qualitative and/or a quantitative investigation of the extracted contamination will occur in consultation with the client.

#### Quantitative determination:

- ⇒ Higher density components in powder samples will be determined by computer tomography and evaluated according to their volume content.

- ⇒ After the selection and the performance of a suitable case-specific separation procedure, the contamination content will be determined gravimetrically

#### Qualitative determination:

- ⇒ After the selection and the performance of a suitable case-specific separation procedure, the contamination fraction is determined by light, infrared or scanning electron microscopy

## **9 Investigation Packages**

### **9.1 PowderGenetics**

This work package includes Powder Genetics WP8.1 to WP8.6 including reporting according to WP10.1. Characterization of the powder in terms of: macroscopic features, chemical composition, determination of elements C, S, H, N and O, water content, particle size and distribution, higher density particles, cavities, geometry, topography and morphology. For this examination, a minimum sample powder quantity of 45 grams is required. Provision of test report documenting the test results including illustrations, spectra and possibly graphical representations.

### **9.2 Particle Ident**

This work package includes the preparation of particles bound in oils or filters for particle characterization. For the investigation, a scanning electron microscope with an energy-dispersive micro-area analysis is used. After preparing a sample survey, the main particles (maximum 5) are determined in their elemental composition and examined both morphologically and topographically. Provision of test report documenting the test results including illustrations, spectra and possibly graphical representations.

### **9.3 In-situ 2D X-ray inspection**

Triggering technical systems (for example electrical connectors, sensors, switches, mechatronic systems, etc.) electrically, mechanically and/or thermally for visualization purposes of inner movements and functions for in-situ experiments. Analyzing flow behavior of internal cooling structures by exposing the test specimen to fluids. Results are documented in video recordings.

## **10 Reporting and consulting**

### **10.1 Material and failure analytical services**

Provision of a standardized IABG report summarizing all relevant results - including diagnosis and evaluation of the damages (failure mechanism), the discharge of conclusions (root cause) and suggestions for improvement (failure prevention) according to the VDI- guideline 3822 (basics and implementation of failure analysis).

Issuing test protocols or test reports for the realized material analysis, evaluation of results, transcription of customized needs and requirements.

### **10.2 Material and failure analytical expertise**

Access to highly qualified pool of experts (internal and external) for customer-specific requirements.



## **11 On-site service**

### **11.1 Ambulant metallography / On-site expertise of failures**

Ambulant metallography including equipment and on-site-expertise of failures upon request.

If the scope of services agreed in the examination plan changes, this will be recorded in a supplementary list, confirmed by both sides and charged accordingly. Travel expenses are not included and will be charged separately. Place of performance is unless otherwise agreed Ottobrunn.