Technical ceramics: tracking down defects

Terahertz measuring technology for non-destructive testing of materials

Testing ceramic materials imposes high demands on the test procedure concerned. Researchers at Fraunhofer IPM have demonstrated the power of terahertz measuring technology on ceramic test specimens. Differences in thickness, defects and drill holes were clearly detected.

Ceramic materials have a range of specific properties that fundamentally distinguish them from other materials such as metals and hence make them interesting for a range of diverse applications. Traditionally, technical ceramics are used as insulators. In machinery and plant construction, ceramic components or functional ceramic coatings guarantee protection against wear and corrosion. In electronics, technical ceramics are used as carrier plates for integrated circuits and also have applications in machining tools and medical prostheses. Extreme hardness and mechanical strength, resistance to wear and corrosion, low weight, heat stability at temperatures well above 1,000 °C, high electrical insulating capacity and a good physiological compatibility distinguish technical ceramics. One further advantage is that the properties can be selectively influenced by the composition of the raw materials, the forming operation and the sintering process. This structural design, as it were, leads to a defined microstructure that determines the mechanical and

Technical ceramics: Ceramic heat shields protect gas turbines from extreme heat and corrosion.

physical material parameters. At the same time, it also represents a weakness in this class of materials. Ceramics are considered to be brittle. Unlike metallic materials, they are not able to compensate for minor structural defects by means of elastic and plastic deformation. The error tolerance in manufacturing high-performance ceramics is correspondingly low. Consequently, precise and consistent test procedures are demanded which are capable of reliably detecting the pores, inclusions, cracks or inhomogeneities in density.

Established methods of materials testing such as optical, acoustic, magnetic, electrical or electromagnetic techniques can be employed for testing high-performance ceramics provided certain adaptations are carried out (see box). Key factors in selecting the process alongside reliability are speed and ease of integration into the production process. One non-destructive test method for ceramic materials that has so far remained untried is terahertz (THz) measuring technology. Scientists at Fraunhofer IPM have for the first time analysed ceramic samples using THz waves and have to a large extent obtained very promising results in the process.

THz waves: Uniting the benefits of adjacent spectral regions

With frequencies between 0.1 and 10 THz, terahertz waves lie between microwaves and infrared radiation. The corresponding wavelengths range from 30 µm to 3 mm. Terahertz radiation combines the benefits of adjacent spectral regions: high depth of penetration and low scattering while achieving good spatial resolution. Due to its comparatively low energy, THz radiation does not initiate any changes in chemical or biological structures, as opposed to UV radiation or X-rays. It is therefore not harmful to humans. In contrast to visible light and IR radiation, THz radiation penetrates many dielectric materials. As a result, it is possible to make structures on the inside of a three-dimensional body visible. It is important to remember here that electrically conductive materials reflect THz waves. THz measuring technology is therefore unsuitable for analysing solid metals but is consequently all the more suitable for detecting metal inclusions in non-conductive materials, e.g. ceramics or plastics.

Depending on the specimen, measurement takes place in a transmission or reflection geometry. In the transmission configuration, cavities or foreign bodies in the material are detected by means of changed absorption or scattering; alternatively, defects or variations in thickness may be derived from the time delay of the pulse compared with time taken by the pulse to pass through a reference sample. In the reflection configuration, the time intervals between the reflected pulses are used to draw conclusions about the thickness of the layer or the site of reflection, since part of the THz wave is reflected at every boundary surface. In this way, cavities or foreign bodies can be detected by the occurrence of additional pulses. THz tomography is a mapping procedure that evaluates the differences in delay. Lateral resolving power is limited physically by the wavelength of the radiation employed, which lies in the region of 300 µm. Added to this is the component due to the apparatus, which is determined by the imaging of the sample volume onto the detector. The resolving power can be varied within a wide range and is at best only limited by the wavelength. In practice it is therefore possible to detect foreign bodies or defects of the order of one millimetre without difficulty. An analysis of the pulse delay times enables conclusions to be drawn about the layer thickness. The greater the thickness – both physical and optical – of the layer to be irradiated, the longer will be the time delay of the THz pulse compared with a reference value or with the measurement on a thinner position of the sample. With the aid of highly precise pulse delay analysis it is possible to investigate layers with thicknesses of some centimetres at an accuracy down to below 10 µm. Achieved signal-to-noise ratios have values of better than 1000:1.

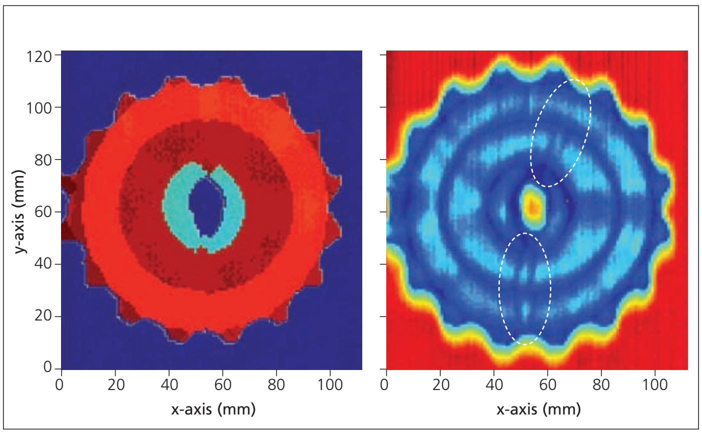

Figure 1: The geometric properties of the sample (thickness arcs) are clearly visible. The transmitted intensity (right) indicates possible structural faults in the actually homogeneous surface.

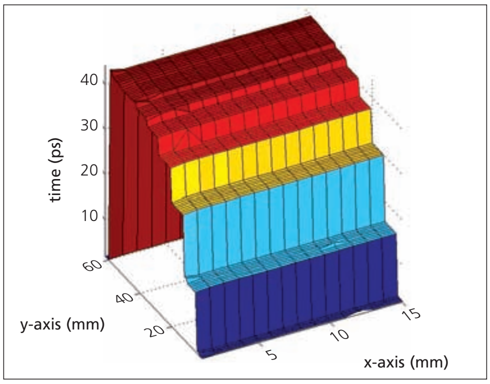

Figure 2: THz imaging shows the 7-part characteristic form of the step wedge. Layer thicknesses of down to 10 µm could be resolved.

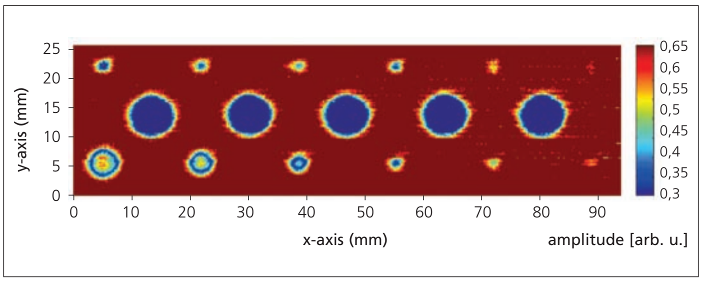

Figure 3: The hidden drill holes in the aluminum oxide plate with a diameter of 1 mm can be clearly seen even at a depth of only 25 µm.

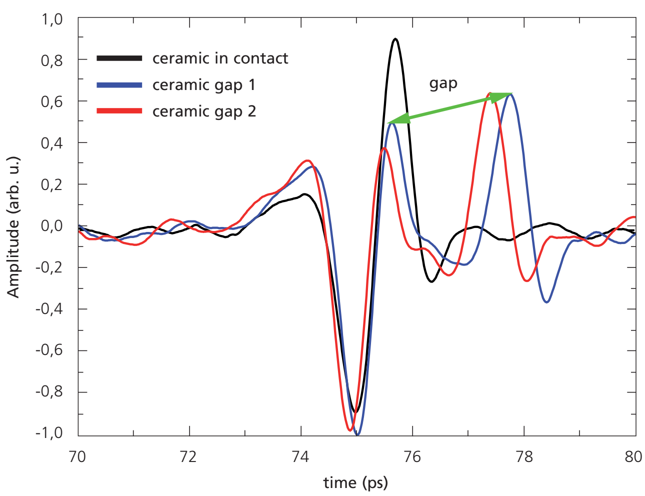

Figure 4: The gap (marked green) indicates an unexpected boundary layer which hints at embedded air.

Thickness, shape and defects clearly detectable

Scientists at Fraunhofer IPM have conducted non-destructive investigations of ceramics relevant to industry provided by Siemens by means of THz imaging. These experiments used various samples to test spatial resolving power in the two lateral directions as well as in the depth axis. Among the materials investigated were an aluminium oxide sample with drilled holes and concealed defects, a step wedge made of aluminium ceramic and a gear wheel made of spinel ceramic. THz measurements were conducted on the samples in transmission or reflection mode in the frequency range of at least 0.1 to 4 THz. Although, the spectral information was present, it was not given any further consideration. It may perhaps be used for a subsequent analysis of active or inactive ingredients. The broadband nature of the measuring apparatus is necessary for the required spatial resolving power and the short pulse length. Samples were positioned in the beam waist, which had a diameter in air of roughly 1 mm, and analysed in a scanning process. Portable or fibre-based systems were also used according to the accessibility of the measuring object.

Evaluation by means of imaging procedures gave good results in terms of demonstrating the geometric properties. In the gear-wheel-shaped sample, for instance, not only the external edge is visible, but the differences in thickness (arcs) and edges can also be clearly seen in the delay image on the left (Fig. 1). Other structures and patterns, which indicate possible structural faults (cracks, possible radial at the top) in the actually homogeneous surface, can be seen in the transmitted intensity on the right (Fig.1).

The 7-part characteristic shape of the step wedge is also well expressed in the various representations. It has been shown that the delay data are the most likely to give information on thickness (Fig.2).

Faults were introduced into the interior of a multi-layered, approximately 2 cm thick aluminium oxide plate and were therefore not visible from the exterior. All drillings can be clearly seen in the various evaluation modes (damping of the THz waves or delay analysis. Even drilled holes with a diameter of 1 mm at a depth of 500 µm and drilled holes with 2 mm diameter at 25 µm depth can be resolved, as shown here in the image of the transmission (Fig. 3).

Similar findings apply to the saw marks made of up to 50 µm in width and 25 µm in depth. Composites made of

several ceramic types or layers can be especially well analysed with a reflection configuration, because in this case a defined reflection occurs at every boundary layer. The position and thickness of the material can be determined via the delay of the pulse. Figure 4 clearly shows the pulse train of the reflection of a ceramic transition (black as the reference). If now an additional boundary layer occurs as an air inclusion, a double pulse structure will appear. The position of the echo (red, blue) enables conclusions to be drawn about the thickness of the air gap.

The measurements performed by Fraunhofer IPM have shown that THz measuring technology is in principle suited to testing inside the bulk of technical ceramics and for measuring the thicknesses of layers. Depending on the material composition, it is possible to reliably measure thickness, shape, cracks and defects in samples that are several centimetres thick. Problems with ceramics might arise due to the presence of typical additives such as pigments or other constituents, e.g. zirconium oxide (ZrO_2). They absorb part of the radiation and may therefore have a distorting effect on the measuring result. Another disadvantage of the measuring procedure is that measurements so far have still been very slow so that, with the present status quo, only measurements of individual pieces in a raster scan are possible – with rates of 0.1 to 200 pixels per second depending on the thickness. THz researchers at Fraunhofer IPM are currently working on multidimensional detection, which will significantly speed up the measurements.

Established non-destructive test methods for ceramic materials:

- **Visual:** Surfaces are examined by means of a microscope, in some cases by using dye penetrants.

- **Sound analysis:** Detection of defects via changes in the acoustic signature of a ceramic component

- **Thermography:** Heating of the component, recording of the temperature pattern at the surface and the layers close to the surface. Detection of defects by means of altered heat diffusion and cooling rate at the surface.

- **Ultrasound:** Detection of defects close to the surface. Scanning of the component at high frequencies (20 to 200 MHz).

- **X-ray computer tomography:** Mapping of the density distribution of section plains yield a reconstituted 3D image. Inspection of complex components with hidden structures possible. At low resolutions, results possible within a period of minutes.

Potential as new NDT procedure

THz measuring techniques offer a number of advantages over the established non-destructive testing methods (see box). They enable measurements to be conducted on the surface and in the bulk of the material. In these techniques THz measurements require no contact, but unlike the similarly contact-free function of X-rays they pose no risks to humans. Neither do the comparatively low-energy THz waves cause any changes in the object under investigation. In principle, THz measurements can be integrated into production processes; one important reason for this is the fact that they are independent of the temperature of the workpiece under test. This wealth of favourable properties makes terahertz measuring technology a promising candidate for non-destructive materials testing.

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