·Home
·Table of Contents
·General

High-resolution radioscopy and tomography for light materials and devices

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New manufacturing techniques and innovative materials, nowadays used in modern product design and fabrication, require elaborated quality control methods by non-destructive testing (NDT). Prominent examples for the needs of high-performance characterization and quality control methods are advanced structured materials (e.g. composites, organic and metallic foams) and microelectronics (e.g. flip-chips, glob-tops). In many cases, the fine structure in those material categories demands the highest spatial resolution achievable. Especially in microelectronics, the recent and upcoming advances in miniaturization will impose the needs for progress in methods of non-destructive testing. The growing number of connections in electronic circuits and components will result in a poor yield if no innovative testing methods would be applied.

The Fraunhofer Institute for Non-destructive Testing (IZFP) develops and deploys innovative testing methods and testing systems for material characterization and the inspection of components.

X-ray methods work non-destructively, impose no need for excessive sample preparation or special sample environment. Depending on the density of the material and the employed X-ray energy, the radiation can penetrate into solid samples to a depth between several microns and several centimeters. Therefore, they enable the inspection of buried or hidden structures, interfaces and defects. There are various kinds of interaction between X-rays and matter. X-rays are absorbed in dependency on the density of the material. At an interface of two materials they are slightly refracted. Due to their small wavelength, which is in the order of the inter-atomic distances, they are diffracted at a crystal lattice. By defects, they are diffusely scattered. X-rays can be used to stimulate fluorescence.

Conventional X-ray radiography has been existing for more than 100 years and is widely applied for the determination of the material distribution in an absorbing object. Via two independent measurements of the incoming (without object) and the transmitted intensities the integral absorption along the propagation path can be determined. For detection of the radiation, conventional X-ray films are still in use. So-called radioscopy employs a two-dimensional pixel detector (e.g. X-ray to visible light conversion by a scintillator crystal and successive recording by means of a CCD detector) instead of the film which allows of simple further processing of the acquired data. Computerized tomography (CT), for instance, consists in the recording of several projection radioscopic images at different rotation angles of the sample (with respect to the beam). In combination with adapted mathematical algorithms, a quantitative three-dimensional image of the absorption coefficient in the sample can be reconstructed. Originating from medical applications, CT can be used for NDT of defects, e.g. inhomogeneities, pores, cracks etc.

Up to now, there have been cases where the conventional absorption radiography and tomography could not be applied for NDT:

- The investigation of objects which do not provide sufficient absorption contrast: This occurs especially in the cases of 1. light, weakly absorbing materials or very thin objects, 2. materials consisting of multiple components with nearly the same absorption coefficients and 3. in the case of high X-ray energies.
- The investigation of objects with a three-dimensional spatial resolution considerably better than 10^3 μm^3 voxel size.

Recent developments in X-ray radiography extend the method towards so-called phase-contrast radiography. This method and the use of synchrotron radiation overcome the limitations described above. Phase-contrast radiography employs the wave properties of X-rays characterized by amplitudes and phases of the wave field. More precisely, phase-contrast radiography may be adequately described by the framework of Fresnel diffraction. Absorption of the sample gives rise to amplitude modulation of an incident wave, whereas refraction due to density variations cause phase shifts related with a slight deviation of the propagation direction of waves.
In this way the phase shifts give rise to interference patterns, which also depend on the distance the wave field has propagated behind the sample up to the detector. Directly behind the sample (at zero distance between sample and detector), only absorption contrast appears. At increasing distance, interference effects generate phase contrast, which in result emphasize interfaces in the radiographs as well as in the tomographic reconstruction.
Recording interference patterns at different distances permits holographic imaging[1] and to reconstruct the three-dimensional distribution of absorption *and refraction* coefficients, giving a precise and high-contrast image of the sample structure. That is why sharp images of the inner structure may be obtained even for transparent samples or those without any absorption contrast. Using synchrotron radiation, the resolution of the images can be up to $<1\ \mu m^3$.
The phase-contrast method is based on a high spatial coherence of the incident radiation, which is provided by small source sizes and large source-sample distances as optimum. Partially coherent radiation may be produced by laboratory microfocus X-ray tubes[2]. Highly coherent X-radiation can be achieved by third generation synchrotrons sources.
In this way, coherent radiation allows in an instrumentally simple manner to extend the radiography and tomography onto samples with structures and defects, which are not detectable by conventional absorption radiography.

The Fraunhofer IZFP performs industrial and scientific research both at conventional laboratory X-ray sources as well as at synchrotron sources and cooperates with the ESRF in providing NDT with synchrotron radiation.
Dedicated beam lines suitable for serial testing are set up at the ESRF. Besides the possibility to use phase contrast, the extremely high intensity of the source, combined with fast electronic detector systems (e.g. the ESRF FReLoN camera[3]) result in sharp images with highest spatial resolution and with low noise level even at short exposure times. The easily adjustable photon energy permits element-specific investigations at absorption edges of the chemical elements constituting the sample. Short exposure times allow to perform *real-time* imaging experiments. To summarize, all of the above mentioned advantages in combination with *effective sample handling* contribute to a cost-performance ratio which puts the relative high beam costs into a favorable perspective concerning *highest quality*, *reliability* and *low overall testing time*.
Synchrotron radiation sources provide a brilliance which is between 6 and 8 orders of magnitude higher than the brilliance of the characteristic radiation emitted by X-ray tubes and up to 11 orders of magnitude higher than their continuous radiation. Consequently, the overall testing time is considerably lower.

Outstanding features of the experiments, which characterize performance, are

- *Highest resolution* at a nominal pixel size of down to $0.4^2\ \mu m^2$ corresponding to a true resolution of below $1\ \mu m^2$ (see, e.g., fig. 3b).
- Utilization of *phase contrast* for the inspection of a) samples providing little absorption (e.g. thin samples and/or light materials), b) samples consisting of a mixture of several similarly absorbing components (see, e.g., fig. 2).
- *Real-time experiments* with a temporal resolution of up to 15 frames per second (see, e.g., fig. 1a).
- *In-situ experiments* by integration of a sample environment for testing under mechanical, magnetic or temperature load (see, e.g., fig. 1a).
- *Element-specific investigations* at the absorption edges of chemical elements.

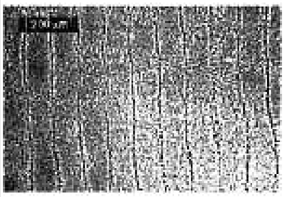

Fig 2a

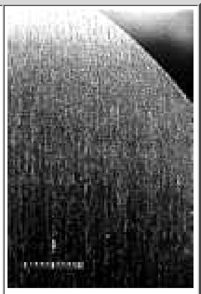

Fig 2b

Fig 2: Radioscopic images of carbon fiber reinforced epoxy.

The experimental setups allow

a. the translation of the samples, e.g. for the scanning of large objects, such as 300mm wafers used in microelectronics,
b. the rotation of the samples, e.g. for laminography and tomography. Samples which are not exceeding the field of view of the used camera can be imaged in one single scan this way. The available high intensity allows reduced exposure times (ranging from several seconds down to some hundredths of a second, depending on the material of the sample and the X-ray energy used), and
c. the integration of sample environment for *in-situ* experiments.

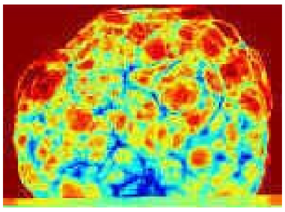

Fig 1a: Radioscopic image of a fully developed metallic foam

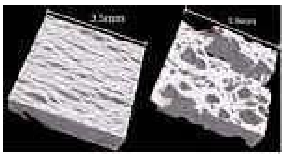

Fig 1b: Al foam at two different stages of the foaming process (ex-situ tomography).3D-renditions of tomographic reconstructions. The red regions

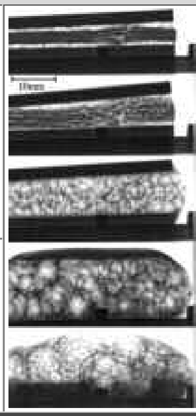

Fig 1b: In-situ real-time radioscopy of a foaming Al sandwich structure. Several stages of the temporal evolution are shown (from top to bottom): appearance of cracks, formation of round pores, melting of the cover sheets, drainage up to the collapse of the foam's structure (see real-time movies on http://www.metalfoam.net/xraymovies.html).

Convincing results have been obtained for a wide field of samples and materials, such as organic and metallic foams (see, e.g., fig. 1: real-time, in-situ radioscopic images and 3D-tomographic reconstructions of aluminum foams), composites, fiber materials (see, e.g., fig. 2: radioscopy of carbon fiber reinforced composites), semiconductor materials and electronic components (see, e.g., fig. 3: wafer, high-performance laser, integrated circuits, flip-chips). The following sections show some examples indicating the high performance of the methods described above.

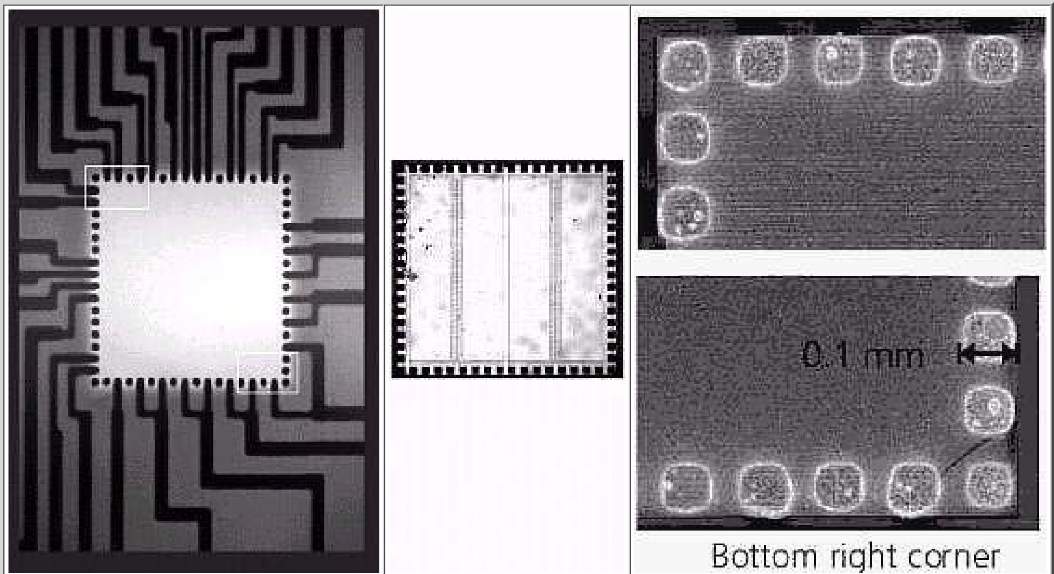

Fig 3a: Reflection light microscopy (before bonding)	Fig 3b: Radioscopy of solder bumps at the marked regions in fig. 3a. The pixel size is 1µm
Fig 3: Examination of flip-chips	

Metallic foams and modern composite materials are supposed to bring vast improvements to individual and public ground transportation which are key elements in modern life. The high stiffness-to-weight ratio of cellular materials and composites could be used to reduce the weight of vehicles and to minimize energy consumption. Moreover, the specific morphology of metal foams dissipates energy very well in crash situations and could therefore be used for improving passive safety.

Fig. 1b shows radiographs which were recorded in real time (in-situ) during foaming of an aluminium foam sandwich structure, i.e. an aluminium foam (low melting AlSi7 alloy) with two dense aluminium face sheets (higher melting wrought alloy). The core layer is essentially a densified mixture of AlSi7 powder with titaniumhydride powder which acts as a blowing agent when the material is heated to its melting point. Upon heating the core layer melts and develops its cellular structure while maintaining its melting temperature for some time. The higher melting face sheets therefore remain solid for some time (1st to 3^{rd} frame). However, if the expanded sandwich is not cooled down after reaching its maximum expansion (3rd frame), further increasing temperatures melt the face sheets and finally lead to a destruction of the foam (4th and 5th frame). It is interesting to note that the foamable precursor material had a crack-like defect in the example shown which, however, dissapeared after foaming. The radioscopic method is therefore very useful in detecting details of the foaming process which are normally obscured.
Fig. 1c displays 3D-renditions of tomographic reconstructions of readily foamed samples (ex-situ tomography). One sees that unlike one might intuitively think the cells are first formed as highly oblate voids which later form fairly round pores. This measurement technique is therefore very useful for investigating the mechanisms of foam formation in early stages which is highly relevant for obtaining better foams of an improved homogeneity.

Fig. 2 displays phase-sensitive radioscopic images of carbon fiber reinforced composites. At internal edges parallel to the incident radiation an increased contrast appears.

Microelectronic devices are spreading into nearly every domain of modern life, due to the steadily advancing miniaturization and the cost reduction as a consequence thereof. Up to now, the inspection of buried structures, i.e. of contacts and interconnections in electronic circuits and components, represents an unsolved task. In order to detect all the defects which compromise the reliability of the entire component group it will be necessary to complement the traditional final inspection by additional inspections. Here we report examples for high-resolution quality control by micrometer resolved and phase-contrast radioscopy and tomography.
Fig. 3 shows results of solder bump inspection in flip-chip interconnections. The bumps consist of a highly absorbing Sn/Pb alloy. Therefore, they are well detectable in absorption contrast. In contrast to radioscopy at conventional sources, high-resolution images at short exposure times are achievable. In our example, high-resolution images with exposure times lower than 1 sec and a resolution of 1 μm^2 pixel size gives evidence of voids of different sizes within the bumps. Fig. 3a shows images of a light microscope of the circuit board and the chip before bonding. The marked regions correspond to the radioscopy images in fig. 3b. The voids are presumably caused by a slight misadjustment of the process parameters in reflow soldering.

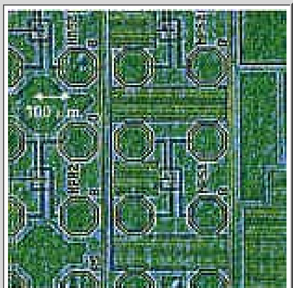

Fig 4: Radioscopic image of an IC

Phase contrast occurs e.g. between Al and Si or Si and SiO_2, which are not distinguishable by conventional absorption radioscopy. Of course, hidden structures would neither be detectable by light microscopy. Fig. 4 displays phase-contrast images of an integrated circuit (IC).
In fig. 5, examples of wire bonded components are shown. Experiments have been carried out inspecting glob-top covered COBs. In the image of Fig. 5a taken perpendicularly to the circuit board's surface, the bonding wires are clearly visible. Fig. 5b shows a 3D-rendition of tomographic reconstruction of the bonding wires and the metallization layer on the board. Fig. 5c (detail) and 5d (overview) indicates a delamination between the glob-top cover and the board which is hardly detectable with conventional testing methods.

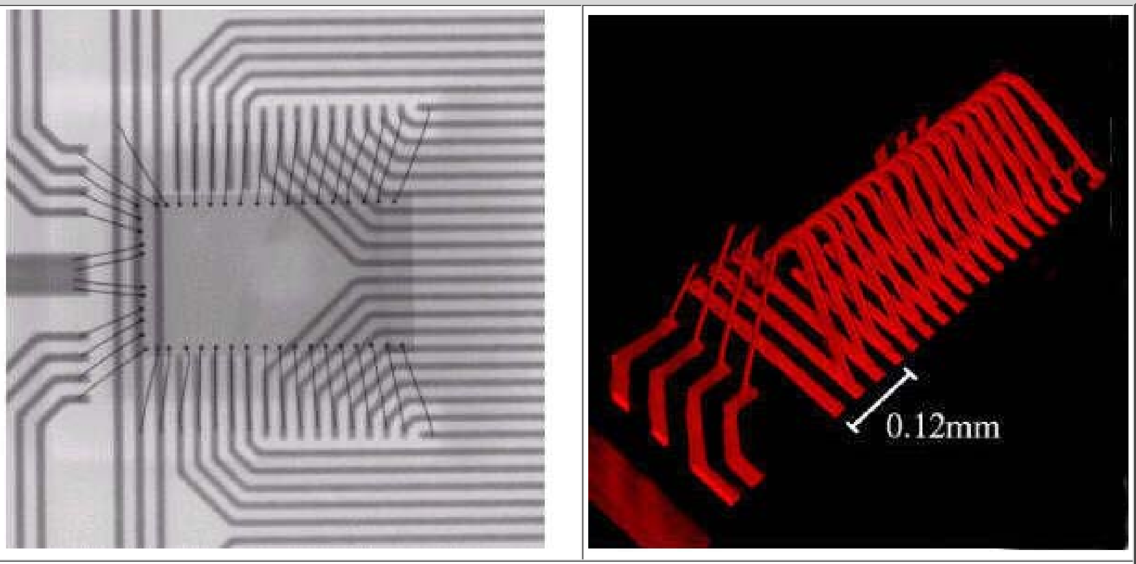

Fig 5a&b: Radioscopy image taken perpendicularly to the circuit board's surface (a), 3D-rendition of a tomographic reconstruction of the bonding wires and the metallization layer on the circuit board on a COB.

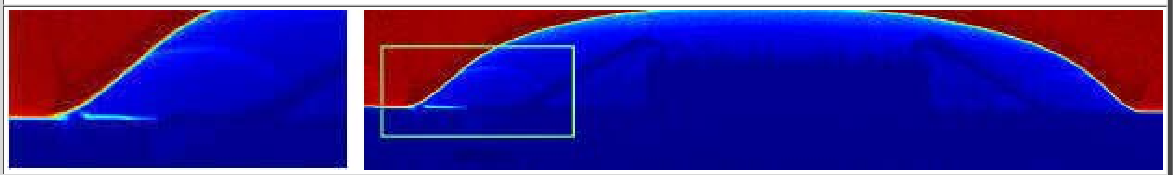

Fig 5c&d: Radioscopy with delamination between glob-top and circuit board: (c) detail, (d) overview

In conclusion we have demonstrated the potential of high-resolution and phase-contrast radioscopy for non-destructive testing of light materials and devices.

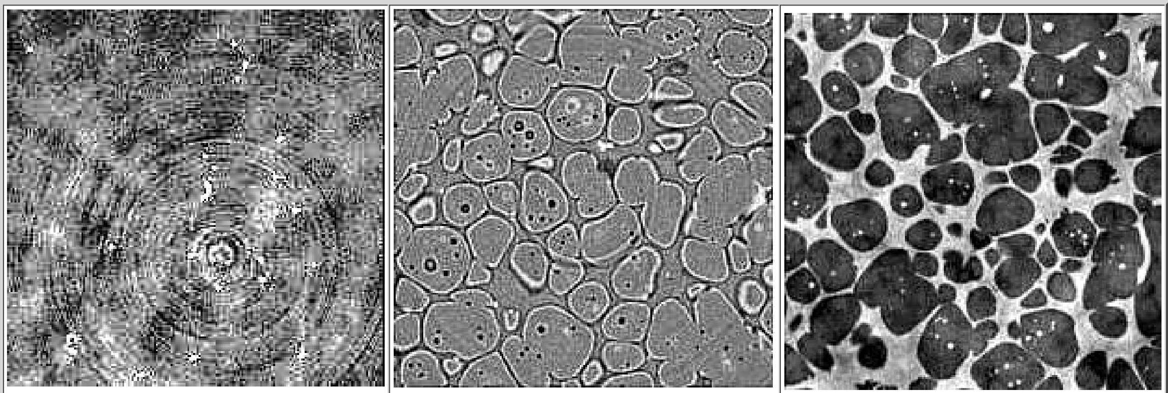

Fig 6: Three tomographic slices of an aluminium-silicon alloy quenched from the semi-solid state, obtained (a) using absorption contrast, (b) using phase contrast and a single propagation distance, (c) using phase contrast and holographic reconstruction based on four distances [4].

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|Home| |Top|