Nondestructive Characterization Technologies for Metrology of Micro/Mesoscale Assemblies*

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Introduction

Micro/mesoscience is an emerging area of science and engineering that focuses on the study of assemblies with small to very small dimensions (~ mm), features and structures (~ µm). Advances in micro/mesoscience will pose new challenges in experimental physics and engineering in general and will lead to new ways of information processing; require new materials with pertinent properties; and lead to new techniques for medical diagnostics and treatment. Research and development of Nondestructive Characterization (NDC) methods to “see” inside or image micro/mesoscale-size materials, components and assemblies will help enable a fundamentally new set of methods that can serve micro/mesoscale science and engineering.

To image the inside of micro/mesoscale assemblies nondestructively requires the use of penetrating electromagnetic waves, acoustic waves, or particles. Micro/mesoscale assemblies typically are of millimeter size and have complex and fragile embedded features or structures that are on the order of a few micrometers. We must be able to nondestructively verify that they are assembled correctly and are not damaged during assembly or have sufficient information to determine if the assembly is acceptable. Therefore, NDC technologies are required that can (1) penetrate into or through a few millimeters of diverse materials ranging from low-density, low-atomic-number foams to high-density, high-atomic-number metals, while providing adequate contrast; and (2) provide spatial resolutions of about a micrometer over a millimeter field-of-view to cover the size of an assembly. These micro/mesoscale requirements define the regions within the electromagnetic (keV x-rays) and acoustic (GHz) spectra and the type of particles (MeV protons) that are applicable to the NDC of micro/mesoscale materials or assemblies.

X-ray, proton and acoustic imaging methods are being developed to characterize assemblies (see Figure 1) with materials that vary widely in composition (low-Z to high-Z), density (~0.03 to 20 g/cm³, i.e., aerogels to fully dense metals), geometry (planar to spherical), and embedded features (joints to subassemblies). Taken together, these techniques will form the basis for a new micro/mesoscale nondestructive characterization capability at LLNL.

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Figure 1. This schematic shows how a notional mm-scale double-shell laser target requires complementary NDC methods to adequately characterize its materials and assemblies. The inner shell is typically a full-density metal or plastic, the outer shell may be plastic or Be and a low-density (10’s of mg/cm³) aerogel is used to support the inner shell. The various characterizations required (top text) are met by the technologies shown (bottom text).

X-Ray Microscopy

How x rays interact with matter depends on the energy of the x rays and the mass density and elemental composition of the assembly. Mass density and elemental composition of the assembly are strongly coupled in the acquired data. X-ray imaging systems can operate in three different modes: radiography, microscopy, and tomography (Martz et al., 2002). The former two result in 2-D projections of a 3-D assembly. They differ in that the short depth-of-field of the microscopy mode results in one plane being in focus with the out-of-focus planes being blurred. In tomography, the 2-D radiographs (projections at near-infinite depth of focus) are reconstructed into a 3-D representation of the assembly. X-ray microscopy and radiography (see Figure 2) are methods to measure the line-integral of the linear-attenuation coefficient, μ, over the x-ray path length, l, (sometimes referred to as μl). In Computed Tomography (CT), many radiographs are acquired and used to reconstruct a 3-D CT model via a computer algorithm. The 3-D CT model is a measure of x-ray attenuation, μ (not μl), in the assembly.
Figure 2. Data acquisition geometries used in industrial x-ray digital radiography and computed tomography imaging. (a) Discrete-beam translate/rotate scanner configuration using a single detector. (b) Well-collimated fan-beam configuration using a linear detector array. This geometry is the most common for industrial and medical x-ray imaging. (c) Cone-beam configuration using a area-array detector. These geometries are also employed for neutron imaging, while for protons (a) is the typical imaging geometry. An example of an x-ray microscope is shown in Figure 5.

To obtain high-resolution (~µm) x-ray images in a practical amount of time (tens of minutes), a high-collection-efficiency x-ray imaging optic and/or an extremely small, bright source should be used. For the former, we are researching a replicated Wölter multilayer x-ray optics system. For the latter, micrometer spatial resolution radiographic imaging of assemblies have been demonstrated using synchrotron x-ray sources.\textsuperscript{1} Given the lack of targets with embedded features available for measurement we exercised the x-ray synchrotron imaging using a target with known surface features. A ruled, 1-mm diameter disk of known construction was selected for x-ray synchrotron computed tomography imaging. The disk was made of polyimide, with a brominated central insert. The surface had a three-dimensional sinusoidal pattern similar to that seen in an egg carton as shown in Figure 3. The ruling was designed to have a 50-µm period, and a peak-to-valley height of 5 µm. The specimen was placed on the rotating stage of the synchrotron radiation microtomography system at beamline 10-2 at Stanford Synchrotron Radiation Laboratory, and scanned in 0.25-degree rotational increments over 180° (Kinney and Haupt, 2002). A double crystal Si (220) monochromator was used to select an x-ray energy of 12 keV (ΔE/E < 10⁻⁴). As a result of the large contrast between the brominated central insert and the unbrominated disk, it has proved difficult to display a topographic image of the entire part in a single image. Therefore, we have chosen to display the insert separately in Figure 4. The surface topography along the datum line is shown in the graph to the right. The period spacing averaged 56 +/- 5 µm and the peak-to-valley height averaged 5.7 +/- 0.9 µm (averaged from line intercepts.

\textsuperscript{1} See papers in Proceedings for the Seventh International Conference on X-ray Microscopy, July 29-Aug.2, 2002, Grenoble, France.
perpendicular to the rule). These measurements are in line with the target (part) specifications. An underlying sinusoidal pattern runs orthogonal to the strong rule pattern seen in Figure 4. This underlying pattern, measured along a trough shown by the datum line in the image to the right in Figure 4, has a sinusoidal nature with a mean peak-to-valley height of 5 +/- 1 µm (averaged from line intercepts parallel to the rule) and a period spacing of 97 +/- 1 µm.

**Figure 3.** Optical micrograph at two different magnifications for a mogel surface machined into a polyimide disk with a brominated central insert used to study Super Nova Raleigh-Taylor instabilities.

**Figure 4.** Left, a CT image of the brominated central insert surface and a graphical presentation of the surface height along the datum line perpendicular to the upper rule pattern. The amplitude is with respect to the mean surface elevation. Right, the CT image of the insert and a graphical representation of the surface height along the datum line parallel with the upper rule showing a wavelength twice that of the upper rule pattern. Only a single peak is fully shown. The negative heights indicate that the surfaces lie below the mean surface elevation.
Other researchers have used grazing-incidence Wöltener imaging optics (Inoue et al., 1989 and Watanabe et al., 2001) with some degree of success, but these optics will be too slow (hours per micrograph) for our application. Xradia\(^2\) and XRT\(^3\), both recent start-up companies, are currently developing x-ray systems based on x-ray zone plates and projection radiography, respectively, for inspecting microchips. Their systems are impressive in their own right tens-of-nanometer resolution at Fields Of View (FOV) of tens of micrometers, but they are geared toward the specific needs of the semiconductor industry, where tens-of-nanometer-size flaws are localized to within tens of micrometers.

A replicated Wöltener multilayer x-ray optics system incorporated into a mechanically stable prototype instrument (Hale, 1999) should be able to meet the micro/mesoscale characterization goals (~1-µm spatial resolution over ~1-mm FOV) (Nederbragt, 2002). Wöltener optics, like other optical systems, entails a tradeoff between spatial resolution and collection efficiency. Since we are satisfied with a ~1-µm resolution, we optimized the collection efficiency for that resolution. Another key to this approach is that the multilayer coating gives it a much better collection efficiency (~100X) over grazing-incidence Wöltener imaging optics. Thus, LLNL is researching and developing an x-ray microscope using Wöltener I replicated multilayer x-ray imaging optics. The twofold objective of this work is to approach 1-µm spatial resolution and operate in a laboratory setting to point the way toward a high-throughput, low-operating-cost micro/mesoscale inspection system.

A super-polished, multilayer-coated, x-ray imaging optic of Wöltener I design is being fabricated. Using a high brightness commercial x-ray source, the Wöltener optic will project x rays through a focal plane in the assembly and onto an imaging plane, providing 12× magnification (see Figure 5). A high-resolution CCD camera, lens-coupled to a scintillator, with 3× optical magnification, will record an image of the focal plane with a detector-element size of about 0.5 µm. Our calculations show that this will take as little as 10 minutes per micrograph image. An 8-keV x-ray source was chosen initially to match low-atomic number and low-density materials of interest. Higher-energy (~60 keV) x rays and matched-energy Wöltener optics will be needed to cover the large range of assembly materials and densities. A “laminate” algorithm will be developed to reconstruct a 3-D image of the assembly from successive focal plane slices through the assembly.

\[\text{Figure 5. Schematic of Wöltener x-ray microscope prototype. It will acquire microscope or microradiographic images using 8-keV x-rays. Its azimuthally integrated collection area has great efficiency potential that allows generation of high contrast images in a practical time frame (10's of minutes).}\]

\(^2\)Xradia, Concord, CA; www.xradia.com.

\(^3\)XRT, Mulgrave, Victoria, Australia; www.xrt.com.au.
Proton Microradiography

Ions, such as protons, in the few-to-100-MeV range lose energy continuously and predictably to matter, predominantly by electron-hole pair production. Therefore, measuring the energy loss of a beam that has traveled through the assembly provides information about the line integral of the areal electron density in the material in a single measurement (see Figure 2a). Repeating this measurement across the assembly thus provides an electron-density map of the assembly. The power of sufficiently-energetic proton beams to penetrate high-atomic-number metals, together with the potential for high-dynamic-range measurements enabled by the roughly linear energy loss mechanism in the material, makes ion beam radiography complementary to x-ray techniques, albeit at the cost of a more involved facility (Gilboy, 1995). High-resolution ion beam radiography is not offered as a commercial or near-commercial capability, however there are several institutions, mainly universities and National laboratories that have accelerator facilities.

Although the interaction of charged particle beams with matter is quite well understood (Bohr, 1913), proton radiography as a high-resolution imaging method is a more recent development (Pontau, et al 1989). In laboratories, proton beams have been used for some time to study the physics of penetration of matter by particles. Protons are useful for high spatial resolution (a few µm’s) imaging, however, this method is hindered by the positional blurring of the protons (Figure 6), known as “straggling.” This blurring is caused by the beam’s strong interactions with the electrical charge distribution of the material through which it travels. Ion MicroTomography (IMT) (Pontau, et al. 1989) measurements were made on a direct drive inertial confinement fusion multilayered target (Antolak, et al., 1992). A focused 2-µm, 8-MeV proton beam was stepped in 0.7-µm intervals to obtain 1121 rays sums or data points per projection and 1761 projections were acquired over 360 degrees. Only 15 proton ions were obtained per ray sum and each CT slice data set required 1.5 hours to acquire. A CT slice was reconstructed and is shown in Figure 7. It is clear that the inner shell is delaminated from the target wall. A line out across the side of the target with the widest separation reveals a delamination or gap of ~ 7 µm. The total electron density is uniform across the shell wall for the slice plane shown.

Figure 6. Schematic of lateral proton straggling in carbon.
A simpler concept than direct imaging by the use of a well focused proton beam (see Figure 2a) was developed even more recently by Doyle et al. (1999) and Wyss et al. (2002 and 2001), and this is the basic approach we intend to implement. This concept employs secondary electron imaging to determine the point of entry for ions impinging on samples under test. Named Ion-Electron Emission Microscopy (IEEM), it circumvents the need for focusing high-energy ions to achieve position accuracy at the micrometer level. Instead of focusing the ion beam and scanning it over the device under test, the positions of the ion impacts are determined by imaging ion-induced secondary electrons at high magnification onto a single electron position-sensitive detector as shown in Figure 8. This enables an accurate location of the proton before it enters the assembly and enables area imaging as shown in Figure 2c for protons; however, it does not overcome proton straggling. We anticipate megahertz-level counting rates that, in turn, should ultimately allow us to accumulate a radiograph of a 3-mm-by-3-mm FOV at a resolution of about one micrometer and a dynamic range of ~100:1 in ~15 min. With the IEEM method, it should eventually also be possible to extract information on chemical species by detection of characteristic x rays (Sakellariou et al., 1997; Antolak and Bench, 1994; Schofield and LeFevre, 1992).
**Figure 8.** Essential components of the proposed proton microradiography prototype. Incident protons/ions (from right) strike an imaging foil, which emits secondary electrons. The electrons determine where the incident proton entered the object. The protons lose energy as they penetrate an object and their energy loss is measured in the ion detector. Signals from the entry of a proton in the foil and its subsequent energy loss are measured in coincidence.

**Acoustic Microscopy**

Acoustic characterization has been in use for decades to evaluate components and materials. Most material characterization takes place in the ultrasonic frequency range, i.e., at frequencies greater than 20 kHz. Characteristics of wave propagation through a solid are determined by reflection, diffraction, or transmission by features such as defects, assembly geometry, material variations, and material attenuation. Two components contribute to material attenuation: scattering and absorption (Krautkramer and Krautkramer, 1990). Scattering accounts for the greatest portion of attenuation in solids, and is caused by material inhomogeneities such as inclusions, porosity, flaws, grain boundaries, grain orientation, and anisotropy. Absorption, resulting from the conversion of acoustic energy to heat in the material, contributes a very small part to the total attenuation in most solids.

There are three common methods of generating ultrasonic frequencies in materials: contact, immersion, and laser Ultrasonic Testing (UT). Both contact and immersion UT require coupling to the assembly by either a couplant or fluid such as water (Birks and Green, 1991), while laser UT is a noncontact method (Scruby, 1990) that does not require couplant (see Figure 9). The different frequencies and spatial resolutions achieved for each of these methods are shown in Figure 10. Immersion UT provides the best Signal-to-Noise (S/N) ratio of the three methods. However, immersion UT is limited by an assembly’s geometry and its immersibility. Laser UT offers rapid, noncontact characterization of assemblies including complex geometries. Laser UT has the lowest S/N and usually requires signal processing to interpret the ultrasonic signals (Thomas et al., 1999 and Candy et al., 1999).
Commercial laser-based acoustic imaging systems (see Figure 10) are available at two discrete frequency ranges: 0.5–25 MHz, called laser UT\(^4\); and 0.1–1 THz, or femtosecond-laser UT.\(^5\) Laser UT has insufficient spatial resolution (~0.2 mm at best) for micro/mesoscale applications. Femtosecond-laser UT has high spatial resolution (~6 nm); however, the penetration depth is inadequate for micro/mesoscale characterization (Richardson et al., 2001). The goal of this research is to achieve acoustic characterization for micrometer resolution of millimeter-sized subassemblies and features. Materials and the geometry of components used in assemblies necessitate the use of a noncontacting technique at frequencies from 100 MHz to 10 GHz. This frequency range is required to acoustically characterize features from 5 to 0.5 µm in size. At this time, laser UT in this frequency range does not exist.

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\(^4\) See ‘LUIS–laser ultrasound inspection system’ developed by Ultraoptec, Québec, Canada; [www.ultraoptec.com](http://www.ultraoptec.com).

The biggest challenge for gigahertz acoustic characterization of micro/mesoscale materials is the depth of penetration. Penetration depth is determined by attenuation of the acoustic wave and is strongly dependent upon the material. For micro/mesoscale acoustic characterization, the attenuation mechanism is different for polycrystalline and amorphous materials. For polycrystalline materials, the attenuation mechanisms are understood. The polycrystalline materials acoustic characterization challenge is to detect features and structures that are on the order of the grain size. For amorphous materials, the attenuation mechanisms are not well understood. Our acoustic research will address these challenges in a three-step approach. First, we need to establish acoustic attenuation parameters at gigahertz frequencies for a variety of applicable materials, both polycrystalline and amorphous. Second, for the amorphous materials, we need to determine and understand the material attenuation mechanisms and incorporate them into existing acoustic models. Third, we need to research and develop a gigahertz laser UT prototype.

**Summary**

Nondestructive characterization imaging technologies are limited in application to micro/mesoscale assemblies. At LLNL, we are researching x-ray, proton and acoustic imaging methods to characterize such assemblies with materials that vary widely in composition (low-Z to high-Z), density (~0.03 to 20 g/cm³, i.e., aerogels to fully dense metals), geometry (planar to spherical), and embedded features (joints to subassemblies). Taken together, these techniques will form the basis for a new micro/mesoscale nondestructive characterization capability.

**References**


