Lothar Spiess, Gerd Teichert, Rene Böttcher, Thomas Kups, Peter Schaaf

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Only the Combination of Different NDT Methods of Material Characterization is the Key to Success

Lothar SPIESS [1,2], Gerd TEICHERT [2], Rene BÖTTCHER [1], Thomas KUPS [1], Peter SCHAAF [1,2]
[1] TU Ilmenau, Institut für Werkstofftechnik, Ilmenau, Germany
[2] MFPA Weimar, Prüfzentrum Schicht- und Materialeigenschaften, Ilmenau,

Contact e-mail: lothar.spiess@tu-ilmenau.de

Abstract. Newly developed, as well as well-known materials have to fulfil requirement profiles for their application. Defects, wrong compositions, and wrong material treatment procedures can have severe consequences in resulting materials properties. Therefore, non-destructive materials testing methods are very important for safety reasons. Nevertheless, for complicated material problems, several complementary non-destructive methods have to be combined in order to archive the all-embracing material characterization. Such methods are described here:

By means of the X-ray diffraction (XRD) methods, phase composition, phase transformations, stress states, and textures can be measured. Sometimes, additional statements about the microstructure are achievable as well.

Material compositions can be accurately determined by X-ray fluorescence (XRF) and glow discharge optical emission spectroscopy (GD-OES). Solutions of the fundamental parameter analysis increase the possibilities of the most analytical techniques and is widely used in XRF analysis and in XRD.

Macroscopic and microscopic structures can be derived with Computer-Tomography (CT). In certain cases, even elemental distributions can be observed, e.g. the distributions of lead in brass material.

To ensure consistent inspection quality, the determination of the mechanical material characteristics such as elasticity module, shear modulus and Poison number is eminent. These parameters should be determined as far as possible non-destructively.

Mechanical material properties are important for the assurance of quality. They can be derived from ultrasonic (US) measurements. With the impulse-echo ultrasonic procedure in diving technology in connection with a special Fourier filtering of the echo signals, these values can also be determined. In some cases, even characteristics of the microstructure can be derived in a fast and non-destructive way, too.

All the mentioned NDT methods and their beneficial combination are exemplified on brass materials for the judgement about materials faults. In addition, purification procedures and the production of historical objects (brass horse muzzle from 1597) can be derived impressively.

1. Introduction

Modern materials exist as alloys, compounds, layered materials. Sometime a special heat treatment performs the properties behavior. To measure the materials behavior, a combination of measurements methods is essential. Sometimes the NDT-methods are more productively and faster than destructive methods. After a useful calibration, these methods can be used for quality management in laboratory and in production.

2. Methods

There exist many non-destructive measurement methods to analyze materials. These are in NDT the well known methods:

X-Ray-Fluorescence Analysis (XFA) [1,7]

Computer-Tomography (CT) [7]

Eddy Current Methods [3,6]

X-Ray diffraction (XRD) [2,3,5,6,7,9,11]

- to analyze phases in Bragg-Brentano Geometry,
- to analyze phases with higher sensitivity to surface layers by grazing incidence geometry,
- to analyze stress by means of stress measurements using $\sin^2\psi$ methods,
- to analyze texture by means of measurement pole figures and
- to analyze nanocrystallinity by means of small angle scattering

Glow discharge optical spectroscopy (GD-OES) [1,2,6,8]

With Glow Discharge Optical Emission Spectroscopy (GD-OES) it is possible to determine the concentration of all elements of the periodic table of the elements up to the ppm-range. The method allows to measurement of the bulk concentration or depth profile. It is based on the continuous sputtering of the material and reaches a death of up to several 100 µm. By using the radio frequency mode the GD-OES is usable for investigations on insulating materials. This caused a dissemination in the academic sector and industry.

The flat sample is located on the cathode, fig. 1. The space inside the glow discharge source is sealed with an O-ring of relative soft material and evacuated. A plasma is initiated by creating an electrical voltage between the cathode and the hollow anode. Argon atoms are getting ionized and accelerated in the electrical field between the electrodes towards the sample. Due to the sputtering process atoms are detached out of the samples surface and migrate to the plasma, where they collide with energetic electrons or metastable Argon ions (Penning like ionization). The GD-OES is based on the detection of the light emitted by atoms in an excited state. The wavelength gives information about the element, the intensity is proportional to its concentration. For depth profiling the device measures the occurring intensity over time via photomultiplier or CCD.

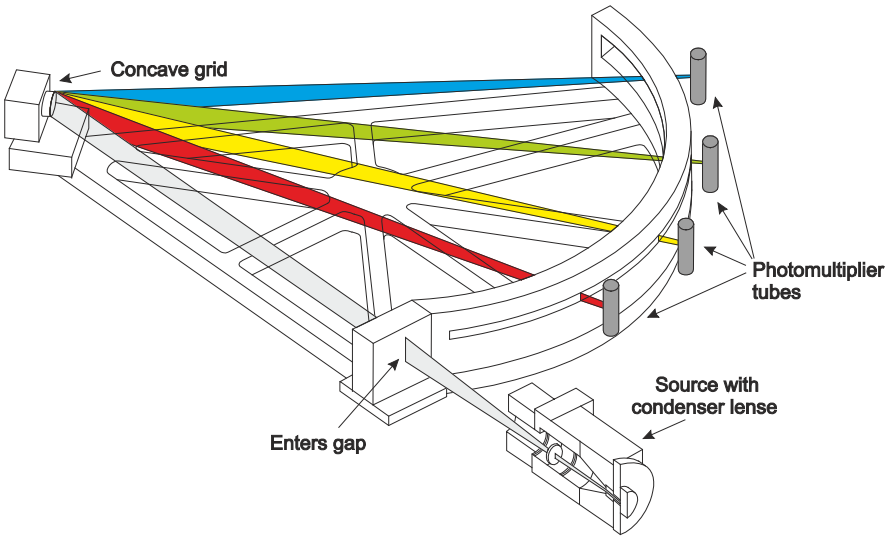

Fig. 1: Layout of GD-OES system

Nanoindentation in preform for fiber optics [4]

Hardness indentations by means of forces in nN range can use to detect the mechanical behavior in grains or along a small layer. Optical and mechanical properties are often coupled. So it was successfully shown, that the necessary change of diffraction index in glass can measured by means of hardness nanoindentation.

Ultrasonics for estimation of materials behavior [10,11]

Ultrasonic is used for investigation on every kind of material and has a general meaning for NDT. Main focus here is the determination of thickness and flaw detection. In our case, it is also possible to estimate mechanical properties of materials, e.g. the Young's modulus E, shear modulus G and the Poisson's ratio μ. By measuring the time delay between two or more echoes, the sound velocity is calculated by using equation 1:

$$c = \frac{2d \cdot n}{t_{i+n} - t_i} \qquad (1)$$

Combining the sound velocity of longitudinal and transverse waves with the density, it is possible to calculate the elastic properties of the sample, equations 2.

$$E = \rho \cdot c_T^2 \cdot \frac{3 - 4\left(\frac{c_T}{c_L}\right)^2}{1 - \left(\frac{c_T}{c_L}\right)^2} \qquad G = \rho \cdot c_T^2 \qquad \mu = \frac{0{,}5 - \left(\frac{c_T}{c_L}\right)^2}{1 - \left(\frac{c_T}{c_L}\right)^2} \qquad (2)$$

When using diving technology and only longitudinal waves, the investigation of layered materials up to several 100 μm is also implementable. To estimate the materials mechanical properties, only a step in the material and the thickness at this point must be given [10]. Apart from this, the structure can be characterized by using the absorption coefficient α, equation 3. There is a dependency of the structure, the used frequency f and the absorption coefficient α.

$$\alpha = \frac{20}{d} log_{10} \frac{p_0}{p(d)} \ [dB] \qquad (3)$$

3. Examples

Brass with lead, mechanical behavior in brass fittings [7,9]

Brass is a well known and in technique often used material. In heating system installations there were used many fittings. The cost pressure leads that in composition were made some changes. It is well known, that the mechanical behavior depends from the composition. Changing of 2% more cheaper copper can increase the material strength – the possibility that brittle fracture are assumed - and the deceasing of plasticity occurs. With XFA it is possible to estimate the composition and with XRD to measure the phases, the grain size and the stress. With Nano-Computer- Tomography, we can find the distribution of 2 – 3 wt% Lead. Lead made the mechanical processing easier, but big agglomeration can be the reason of beginning cracking of the material.

Brass horse muzzle from 1597 [5]

The way of forming (rolling or hammering etc.) to manufacture a brass horse muzzle (art-historical very valuable work from 1597) should be found out within the scope of restoration works. The composition of the brass was determined by means of intensities of energy dispersive X-ray fluorescence analysis (XFA). X-Ray texture test measurements were performed with comparable composition of rolled and hammered brass samples. The different technology to form the brass sheets leads to different texture of grains.
The pole figures and the structure pictures clearly differ between hammered and rolled material. This is seen also the XRD diffractograms in Bragg Brentano geometry. Then the complete horse muzzle was examined in the X-Ray-diffractometer at different places and a texture comparable to hammered tests was found. Therefore, it was assumed that this horse muzzle was made by hammering the brass sheets and no rolling was used at this time.

Aluminum alloys and US [11]

Nowadays there are different specialized alloys, especially in lightweight design. Small changes in composition often have big influence on the mechanical properties. It is possible to estimate those changes by using simulation software like JMatPro* from SENTEC Software Ltd*. This was done for different Aluminum alloys with variated silicon and manganese concentration regarding the Young's modulus. Ultrasonic measurements and tensile testing delivered the result that there is a good accordance between simulated values and ultrasonic results. Even small changes are visible. By using tensile testing this is not possible. The small changes perish from the error. By using Ultrasonic it is feasible test the sample fast, easy and nondestructive. The results are also much more precise, fig. 2.

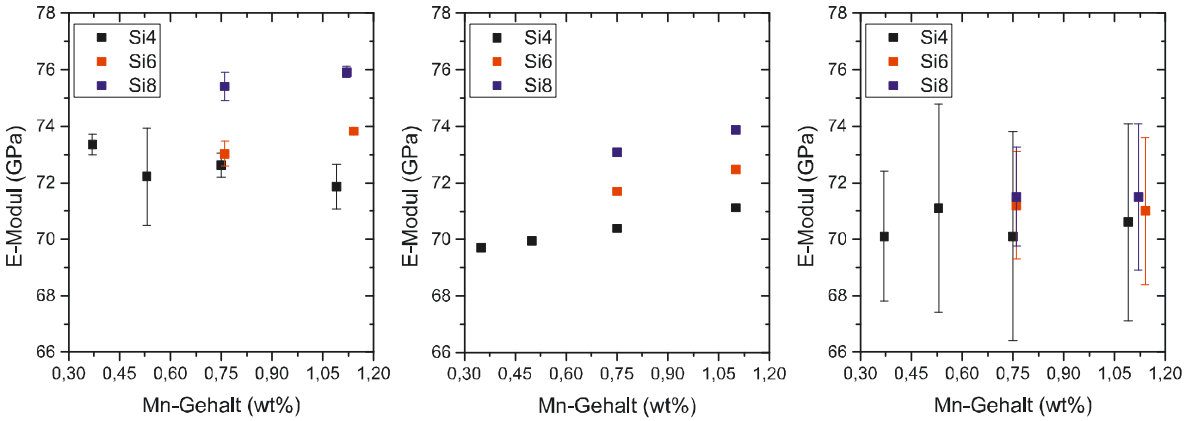

Fig. 2: Measurement and simulation of the Young's modulus E dependent on the Si- and MN-concentration
a) by using ultrasonic b) by simulation with JMatPro c) measured via tensile testing

Gold falsification [10]

A thin tungsten sheet of 0.44 mm was coated with a layer of gold of 30 µm. By using XRF (30 kV, tungsten anode) it was found only gold. The tungsten sheet behind was not detectable. The Gold layer is too thick for this RFA-method. Ultrasonic measurements of sound velocity in the coated area showed the detectability of the tungsten core by comparing the sound velocity of pure gold and the measured one of the faked ingot, table 1. The bigger error in estimation velocity is also an advice for the falsification, because of the scattering of the sound waves at the interface.

Tab. 1: Estimated and measured ultrasound velocity

material / sample	density [kgm^{-3}]	d [mm]	c_L [ms^{-1}]
pure Gold (literature)	19300		3240
Tungsten	19250	0,44	5261 ± 25
"Gold brick"	19270	0,50	5236 ± 257
Dental Gold (750) (75wt% Au, 5wt% Cu, 4wt% W, 15wt% Ag)		1,29	3152 ± 4
Gold alloy (333) (33wt% Au, 44wt% Cu, 8wt% Zn, 15wt% Ag)		0,83	4376 ± 28
Gold alloy (333) (33wt% Au, 39wt% Cu, 5wt% Zn, 22wt% Ag)		1,26	4410 ± 10

The measurements showed that XRF is not able to verify the authenticity of gold ingots. The heavy atoms cause a low penetration depth of the radiation. Ultrasonic is able to realize a nondestructive testing of ingots or real Gold bars. By using calibration curves, it is also imaginable to determine the gold quantity in those alloys. The composition of Gold alloys was estimated by XFA.

4. Conclusion

With this enumeration of methods and examples from the materials technology, it is shown that complicated materials characterizations are only possible by a combination of several complementary NDT methods.
Besides, quicker and more reliable measurements with NDT methods are possible if calibrations and sample access is partially possibly destroying procedures.

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