MICRO STRUCTURAL ANALYSIS OF AlSi6Cu4 USING QUANTITATIVE COMPUTED TOMOGRAPHY METHODS

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1. Introduction

Today, microstructural analysis of metal alloys is usually done by preparing cuts through the object and subsequent light-optical-microscopy (LOM) or secondary electron microscopy (SEM) examination. Initially, this destructive method allows only 2-dimensional cut views, while a 3-dimensional volume generation involves the time consuming and possibly inaccurate preparation of multiple cuts and the following fusion to a 3-dimensional data set. Upcoming new methods like focused ion beam (FIB) tomography allow already a user friendly, but still time consuming 3D analysis of microstructure on a very fine scale, but the investigated volumes are still very small.

2. Computed Tomography

2.1 Micro Computed Tomography (µCT)

Recent developments in the field of X-ray imaging, including detector and X-ray production technologies, allow an increasing spatial resolution. Especially micro focus X-ray tubes with a focal spot size of approximately 1 micrometer in combination with high geometrical magnification allow 3D computed tomography (CT) volume images with voxel sizes of less than 1 micrometer. Actually this method allows the investigation of microstructures down to a size of a few microns in a representative volume of about $(2\ mm)^3$.
From this reconstructed volume, information characterizing the microstructure like dendrite length or secondary dendrite arm spacing can be evaluated independent of the probe orientation. Using this 3D CT Method, the composition of the alloy specified by the distribution of the chemical phases can only be evaluated by a relative grey scale value resulting from different attenuation coefficients. In general, this does not allow quantitative conclusions of the material under examination.

2.1 Quantitative CT

As the attenuation coefficients are not only element specific, but also energy dependent, energy sensitive methods provide the possibility to obtain quantitative information. Comparable to common analysis techniques like the energy dispersive x-ray fluoroscopy (EDX) the dual-energy method provides information about the elemental composition and the physical density of the specimen. This quantitative extension, the larger measureable volume size and the adequate achievable resolution suggest CT for application in metallography.

The 2X (dual-energy) approach is based on a method from medical CT imaging also known as rho-Z-projection (Heismann et al. 2003). This method employs two measurements of the object using different X-ray spectra and yields the effective atomic number and mass density of the specimen.
The 2X CT method requires a very accurate characterisation of the components used in the CT machine. In particular, this is the spectrum of the X-ray tube for the different acceleration voltages including the tube side filtering and the spectral detector sensitivity. In this case, these data was obtained by a Monte-Carlo Simulation [1]. Furthermore, it is necessary to correct for beam hardening artefacts. This is done by applying a linearization curve generated by the method in [2]. As this method can only be applied to one material, it

is applied to a region containing only aluminium.

3. Results

3.1 µCT

The µCT were performed on an Al alloy typically used for high load and heat stressed automobile components. Referring to the well known chemical composition of this alloy, previously analysed by SEM-EDX on a representative layer, several phases can be assigned to a constituent by their grey level displayed in the reconstructed volume. The phases Al-Al2Cu, ß-Al5FeSi and Mg2Si can be distinguished from the primary α-Al phase quite well. But since the detection capability depends on the difference between the specific absorption coefficients and the X-ray energy level, the recognisability varies. Especially when focusing on the Mg2Si phase in the α-Al the limited contrast is obvious.

A comparison of the CT results to a LOM investigation of one and the same layer of the alloy shows the difficulty when resolving materials with nearly the same absorption properties. The Mg2Si phase is nearly existent in the CT cross-section.

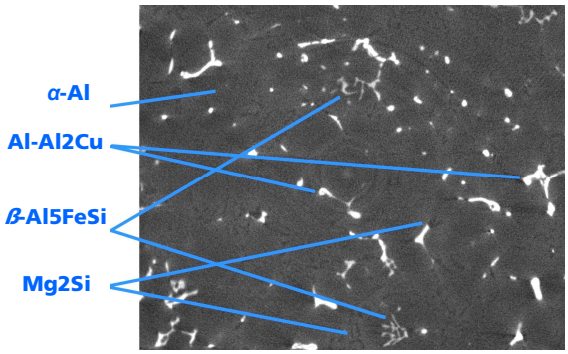

Fig. 1:Microstructural investigation using a standard 3DCT reconstruction of a AlSi6Cu4 alloy.

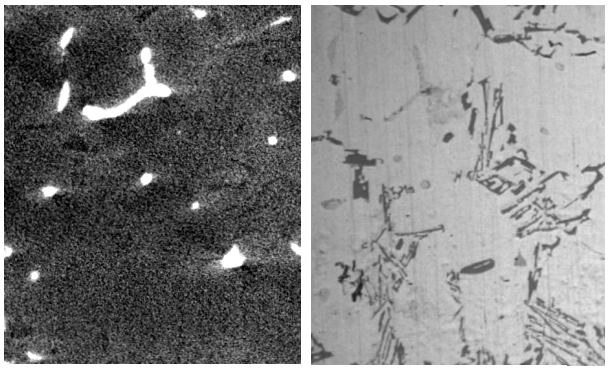

Fig. 2:Comparison of a CT(left) and LOM(right) investigation on one 2D cross-section

On the other side constituents who are hardly visible by LOM without special etching treatment are visible in the CT very well due to their specific difference in absorption.

Since the application of low energy is needed to reach highest contrast, the results are afflicted with a beam hardening which results in an inhomogeneous grey value distribution across the volume. By a linearization [2] this effect can be corrected without a significant loss of information between the structures with low absorption differences.

3.2 2X CT

To demonstrate the 2X-CT method, a macroscopic phantom with known properties was used. It is a aluminium (Al) rod containing inlays of pure titanium (Ti) and copper (Cu). The rod has a diameter of 20 mm and the inlays have a thickness of 0.6 mm (Cu) and 0.9 mm (Ti). The length of the inlays is depending on the position in the object, in the images shown (**Fig 1** and **Fig 2**) it is approximately 2.2 mm for both. The atomic numbers Z and mass densities ρ of these elements can be found in **Table 1**.

Element	Z (atomic number)	ρ (mass density)
Al	13	2.7
Ti	22	4.5
Cu	29	8.9

Table 1: Atomic number and mass density of the elements contained in the Phantom

The CT equipment used for the measurements of the phantom are an Yxlon FXE 225.45 (directional) as an X-ray source and a PerkinElmer XRD 0820 AN15 detector. The two data sets were taken at 120 kVp with 0.5 mm Ti filtering and 220 kVp with 2 mm Cu filtering.

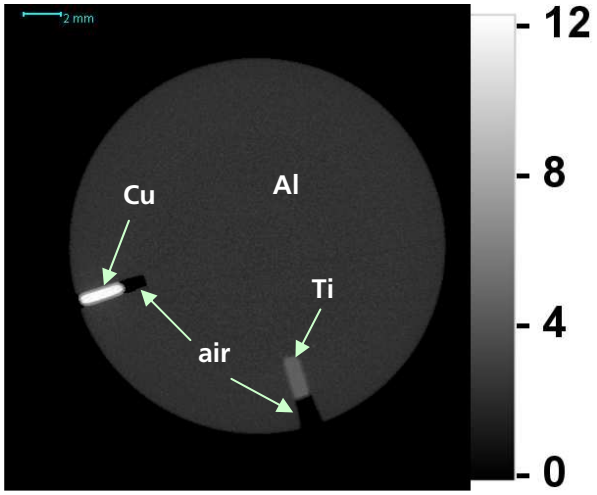

Fig 1: The density image of the phantom. The unit is the physical mass density.

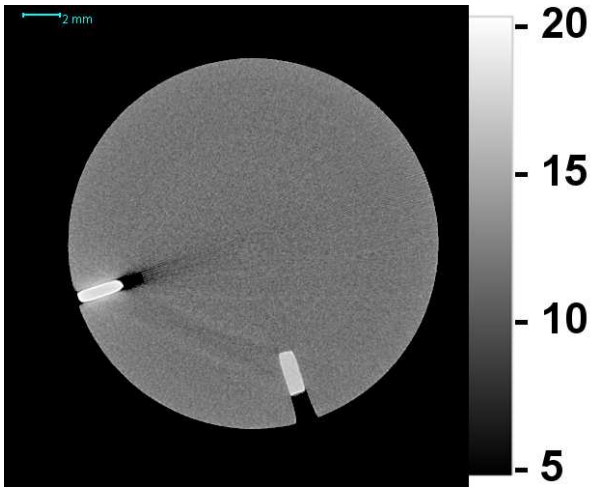

Fig 2 The atomic number image of the phantom. The unit is the effective atomic number Z.

Fig 1 and **Fig 2** show the images of one layer of the 3D data set resulting from the 2X CT. In Fig 1, only the physical mass density is displayed, while Fig 2 shows an images of the distribution of the atomic number Z. The average reconstructed mass density of the aluminium rod is 2.5 g/cm^3, which is in good accordance with the real mass density of aluminium of 2.7 g/cm^3. The atomic number of aluminium is 13 and it is reconstructed in good accordance as 11.8. The density of the titanium content is reconstructed very precisely as 4.5 g/cm^3 and the atomic number around 17.
In the atomic number image, a cupping effect is visible in the copper region, i.e. the value on the rim of the region are larger than in the inner part. In that part, the reconstructed value for Z is around 18, which is clearly too low. In the rim part, with values up to 21, it comes closer to the actual value of 29 for copper.

While the Z value for copper is too low, the density for the copper region is to high. In the rim region, it is very good at approximately 9.0-9.5 g/cm^3, but in the centre region it is clearly too high with values up to 11.8 g/cm^3.

Element	ρ in g/cm^3 (real)	ρ in g/cm^3 (measured in 2X)
Al	2.7	2.5
Ti	4.5	4.5-4.6
Cu	8.9	9.0-11.8

Table 2: Reconstructed mass densities

Element	Z (real)	Z (measured in 2X)
Al	13	11.8
Ti	22	16-17.5
Cu	29	18-21

Table 3: Reconstructed atomic numbers

4 Conclusion and Outlook

A high resolution µCT of a AlSi6Cu4 specimen has been presented, showing the applicability of µCT for 3D micro structural analysis. With respect to the actual technical and physical limitations the µCT cannot replace common metallographic methods. But for a wide application field the benefits of a 3D investigation can be used yet.

The quantitative 2X CT method has been applied on a macroscopic phantom, yielding effective atomic numbers and mass densities. For aluminium and titanium the reconstructed values are pretty close to real ones. However, the reconstructed values for the regions containing copper are less convincing. As cupping artefact is visible there, this might be a result of beam hardening, which is also a function of the atomic number. Beam hardening is corrected only for the aluminium part while copper (Z=29) is clearly separated from aluminium (Z=13) in terms of Z. An other reason for the observed inaccuracies may be scattering of visible light in the scintillator of the X-ray detector, which is not considered at the moment.

In future, the 2X CT shall be applied to the µCT. The very accurate spectral characterisation of the X-ray components needed for 2X CT are currently being done in our group. Furthermore, a complete simulation of the imaging setup would allow the full control of all parameters and effects, in turn allowing the identification of all factors influencing the system.

The further development of the technical components and introduction of new methods like the 2X CT can help establish the CT method for metallography especially in the field of material science.

References

[1] J. Giersch, J. Durst; "Monte Carlo simulations in X-ray imaging", Nuclear Instruments and Methods in Physics Research Section A, Volume 591, Issue 1, 11 June 2008, Pages 300-305

[2] US patent 6975697, S. Kasperl, U. Hassler, I. Bauscher, S. Schroepfer, "Apparatus and method for establishing a correction characteristic curve for a reduction of artefacts in tomography", filing date 06/04/2004