Neutron Diffraction Stress Measurement in an Al-Si Casting Alloy

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Metal matrix composites (MMCs) comprising a soft matrix and hard inclusion particles typically have a high elastic modulus and high strength. The basis for these attractive mechanical properties lies in the details of microstructure (i.e., the size, shape and distribution of the particles), the micromechanical interaction of the matrix with the particles, and the nature of the particle/matrix interface. One of the quantities important for understanding the properties of the composite is the residual stress since it affects the yield stress and the early work-hardening rate \(^1\).

The stresses in composites (two-phase or multi-phase) are often classified as macro and micro \(^2\). Macrostresses are long-range residual stresses which arise from differential thermo-mechanical treatment of different parts of the body. Macrostress can be successfully measured by mechanical methods such as hole-drilling or a variety of layer removal methods, all of which are destructive. Microstress, in contrast, exists on the scale of the particles, grains and other features of the microstructure (e.g. defects) and cannot be measured in the same way as for macrostress.

Radiation diffraction techniques (x-rays and neutrons) are essentially the only non-destructive ways for measuring macrostress. This is because, in diffraction mode, neutrons (or x-rays or electrons) interact with the crystal lattice on the scale of the inter-atomic plane distances and can, therefore, measure the lattice strain. The strain resolution for neutron diffraction is \(\sim 10^{-4}\) which corresponds to a stress resolution in Al alloys of \(\sim 1\) MPa (although, in practice, this stress resolution is hard to achieve). At the same time, neutron diffraction can measure microstress since they can simultaneously measure lattice spacings for the different phases in a composite. Importantly, neutrons have the remarkable property of high penetration into the material (half-attenuation depth \(\sim 60\) mm for Al) exceeding by many orders of magnitude that for other radiation (\(~ 50\) \(\mu\)m for x-rays and \(~ 10\) nm for electrons). This allows measurements to be made that are unaffected by free-surface effects; further, since the volume of material that can be probed is as small as 2x2x2 mm\(^3\) (for Al alloys) stress distributions can be measured at mm-scale resolution (for more strongly scattering elements the volume can be reduced to \(1/4 \times 1/4 \times 1/4\) mm\(^3\)).

In this paper we report stress measurements on an Al-7Si-0.4Mg casting alloy to illustrate the capabilities of neutron diffraction for a two-phase MMC. In this case study, phase stresses were measured, from which micro- and macrostresses were extracted. The existence of a microstress in the particles, arising from the mismatch due to the difference in thermal expansion coefficient between the two phases, is also demonstrated.

Material

The material is the commercial Al-Si-Mg alloy A356 (6.6Si, 0.4Mg, 0.05Fe, 0.08Ti wt%) with 0.02 wt% Sr added to modify the shape of the eutectic Si particles. A sand-cast plate (140 x 160 x 25 mm\(^3\)) was made and 25 mm slices were cut from it and solution heat-treated at 540°C for 6 hours. Then they were cold-water quenched and aged at 170°C for 6 hours. The microstructure comprises dendrite colonies \(~ 0.8\) mm in diameter and inter-dendritic eutectic Si particles with a diameter of \(~ 3\) \(\mu\)m (Figure 1). The volume fraction of the Si phase was measured by neutron diffraction phase analysis as 0.074, close to the calculated value of 0.062.

The yield strength of the alloy was 287 MPa and Young’s modulus was 75 GPa. Test samples with a gauge diameter of 12 mm were machined out of the slices for the neutron diffraction stress experiment.

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Neutron diffraction measurements

The experiment was carried out on the stress diffractometer KOWARI on the OPA research reactor at ANSTO. Stresses were measured in both phases, Al and Si. Although both Al and Si are weak neutron scatterers, measuring stress in the Si phase was especially challenging due to its small volume fraction. Nevertheless, the instrument setup allowed measurements in the Si particles to be done within a reasonable time (typically 20 minutes) with a spatial resolution of 2 mm in the radial direction. A neutron wavelength of 0.1577 nm was used to measure diffraction peak shifts in the Si(422) reflection. Lattice spacings of the reflection in three principal directions, radial, hoop and axial, were measured across the specimen diameter by suitably orienting and translating the sample with respect to the neutron beam.

A standard Si powder sample was used to measure the Si(422) lattice spacing in the unstrained (zero stress) condition: this was used as a baseline for the measurements of the strain in the Si particles.
Despite the high volume fraction of the Al dendrites, the stress measurements in this phase are not straightforward. This is because the large grain size made getting statistically reliable measurements a major issue. The grain size problem was tackled by averaging the data over multiple equivalent radial positions in the sample by rotation and translation. That is, the diffraction peak shifts were measured in the same radial position and the same directions as for Si but the specimen was rotated so that measurements were made over an entire 360° rotation so as to sample many dendrites.

**Stress analysis: separation of macrostress and microstress**

While the experimentally measured quantity is the diffraction peak position, strain can be calculated if the stress-free lattice parameter is known and then stress can be derived using Hooke’s law and the elastic constants (the so-called diffraction elastic constants) [3].

Stress analysis in a two-phase material assumes deconvolution of the measured phase stresses, $\sigma_{\text{tot}}^{\text{Al}}$ and $\sigma_{\text{tot}}^{\text{Si}}$, into macrostress and microstress:

\begin{align}
\sigma_{\text{tot}}^{\text{Al}} &= \sigma_{\mu}^{\text{Al}} + \sigma_{\mu}^{\text{Si}} \\
\sigma_{\text{tot}}^{\text{Si}} &= \sigma_{\mu}^{\text{Si}} + \sigma_{\mu}^{\text{Al}}
\end{align}

(for the sake of simplicity, tensor indices are omitted). Macrostress is the long-range stress that would be found in a homogeneous (single phase) material and it originates from differences in the local plastic and thermal history of a body. To extract the macrostress from the experimental data the rule-of-mixtures applies:

\[\sigma_{\mu}^{\text{Al}} = (1 - f)\sigma_{\mu}^{\text{Al}}_{\text{tot}} - f\sigma_{\mu}^{\text{Si}}_{\text{tot}}\]

Microstresses vary on the scale of individual grains or second-phase particles. They occur in multi-phase materials that have differential elastic, plastic, and/or thermal properties. In single-phase polycrystals they are significant if the material is elastically anisotropic. They are balanced in any macroscopically large volume

\[\sigma_{\mu}^{\text{Al}} - f\sigma_{\mu}^{\text{Si}} = 0\]

and, as noted in the Introduction, cannot be studied with macroscopic methods.

**Results and discussion**

The experimental results for both the macro- and microstress distribution are shown in Figure 2, in which the stress measurements have been made across the 12 mm diameter of the cylindrical specimen. An obvious feature of the data, Figure 2a, is the small macrostress. This is expected in a qualitative sense since (a) Al has a high thermal conductivity, and (b) the specimens were cut from larger bars so that the initial residual stresses were partially relieved. It is often assumed by investigators working in the field of...
Al-based MMCs that the quench stresses are negligible in such small specimens. Our data confirm that they are small but, nevertheless they are not negligible, having the classic features of compression at the edge of the cylinder and tension in the interior. Detailed analysis of the stress distribution usually requires finite element models\(^4\) and this has yet to be done for the present experiment.

A second result is that microstresses are also significant in the composite (Figure 2b). The microstresses in the Si particles of about \(-180\) mPa are an order of magnitude larger than the \(-15\) MPa in the Al dendrites, this partition being determined by the ratio of volume fractions (0.06 for Si and 0.94 for Al) and the requirement that the overall weighted microstress is zero (equation 3). The fact that both Si and Al have isotropic coefficients of thermal expansion, coupled with the elastic isotropy of Al, leads to the expectation that both the Al and Si microstress should be hydrostatic, \(\sigma_{\text{micro}} = \sigma_{\text{radial}} = \sigma_{\text{axial}}\). Figure 2b generally corroborates this assumption. However, to answer definitively the question as to whether the Si microstress is in some degree non-hydrostatic and/or varies with radial position requires further investigation.

These results can be used to test micromechanical models. For example, Eshelby-type calculations suggest that for the Al-Si composite to acquire the observed microstresses, the corresponding temperature change must be \(-120^\circ\text{C}\) \(^3\) and this could be tested by heating the casting to \(120^\circ\text{C}\) above room temperature, when the microstresses should vanish and the mechanical properties (yield stress and work-hardening rate) should be noticeably different. This is the subject of our ongoing research.

In this case study it has been demonstrated that neutron diffraction is capable of measuring stresses in phases with a small volume fraction, \(-0.06\), and that they can be resolved with very high spatial resolution, e.g., 2 mm. Stresses can be measured with an accuracy as good as \(-5\) MPa that allows capturing very subtle stress distributions and treating them quantitatively.

Conclusions
In this case study it has been demonstrated that neutron diffraction is capable of measuring stresses in phases with a small volume fraction, \(-0.06\), and that they can be resolved with very high spatial resolution, e.g., 2 mm. Stresses can be measured with an accuracy as good as \(-5\) MPa that allows capturing very subtle stress distributions and treating them quantitatively.

References

Figure 2: Experimentally obtained macrostress (a) and microstress (b) profiles. The dotted lines in (a) are drawn for only for visual reference.