

Uncertainties in the Measurement of Partially Recrystallised Microstructures

Summary

This measurement note is concerned with the quantification of uncertainties associated with the measurement of partial recrystallisation in a Ni base alloy. Following an interlaboratory exercise on partially recrystallised Nimonic 901 supported by DTI, industry and academic participants it was concluded that:

the uncertainties were at least $\pm 20\%$ for volume fraction, $\pm 40\%$ for recrystallised grain size and $\pm 100\%$ for unrecrystallised grain size.

The principal reason for these large uncertainties is the lack of an agreed method for discriminating between recrystallised and unrecrystallised regions.

Preliminary experiments with a new technique, orientation contrast imaging (OCI), indicated that this could provide a more unambiguous method for discriminating between the two regions of interest.

It is recommended that:

the use of OCI as a generic tool for the measurement of microstructural features is developed further through comparison with reference measurements on a range of engineering materials.

B Roebuck

October 1996

General Background

Metal working industries are developing models for process control that require input data which correlate stress, strain, strain rates and temperature with micro-structural changes. These models will enable quality engineered products to be manufactured with targeted micro-structures. It is important to understand what uncertainties are associated with measurements of various microstructural features so that appropriate sensitivity analyses can be applied to the models.

In Ni base alloys, following hot deformation, products generally contain a mixture of recrystallised and unrecrystallised grains. The properties of the alloys are very dependent on the extent of recrystallisation. Consequently, process models must be able to predict how recrystallisation relates to the applied loads, shape changes and deformation rates during metal working.

Recrystallisation Measurements

To quantify typical uncertainties with current in-house measurement procedures, an interlaboratory exercise was performed on a hot deformed partially recrystallised Nimonic 901 sample. Seven UK organisations from industry and academia participated. This summary is taken from a full NPL report of the exercise [1]. The measurements were made on directly polished sections or photocopied micrographs taken from a longitudinally sectioned axisymmetric testpiece of Nimonic 901 after deformation at 1000°C to a strain of 0.5 at a strain rate of 1 s⁻¹. Measurements were made of volume fraction recrystallised, V_R , recrystallised grain size, d_R , and unrecrystallised

grain size, d_{uR} .

Partially recrystallised Nimonic 901 following hot deformation.

Relevant Standards

There are four ASTM standards which have relevance to the exercise, E112 (Grain size), E562 (Volume fraction by point counting), E1181 (Duplex grain sizes) and E1382 (Grain size using image analysis). The method of measurement was left to the choice of each participant from line intercepts, point counting grids or circle intercepts.

Microstructures

The Nimonic 901 samples combine a mixture of small equiaxed recrystallised grains, frequently containing twins, and large elongated unrecrystallised grains. The presence of twins made it difficult to identify grain boundaries unambiguously and it was not always evident which grains were recrystallised or unrecrystallised, since there are no specified guidelines. The choice is arbitrary and was left to the operator.

Results

Uncertainties are classed as Type A (conventional statistical analysis) or Type B (estimated from errors in the measurement process, ie magnifications, etc). For this exercise Type B uncertainties were ignored, as were correction factors required by current guidelines for uncertainty estimation from standard deviations obtained on a small population of results. A good document to consult regarding uncertainties in testing is published by the British Measurement and Testing Association (BMTA) - "Estimated Uncertainties in Testing", BMTA, PO Box 101, Teddington, Middlesex, TW11 0NQ.

The interlaboratory measurements resulted in 95% confidence limits (± 2 standard deviations) of about:

Volume fraction, Recrystallised, $V_R \pm 20\%$

Grain size, Recrystallised, $d_R \pm 40\%$

Grain size, Unrecrystallised, $d_{uR} \pm 100\%$

For one of the test pieces examined the uncertainties given above were approximately halved, to $\pm 10\%$ for V_R and $\pm 12\%$ for d_R for measurements made by laboratories more experienced in examining these types of structures.

For volume fraction measurements, image analysis gave lower values than point counting. In turn point counting gave lower values than linear intercepts.

The large variation in the measured size of the uncrystallised grains was partly due to the lack of a specified method for measuring grains with large aspect ratios (ie horizontal, transverse, random) and partly due to the difficulty in identification.

Alternative Method

Characterisation of partially recrystallised structures is not standardised and is based on arbitrary criteria. Unambiguous identification methods would improve the accuracy of the required measurements. A possible technique is Orientation Contrast Imaging (OCI). These images, obtained in a scanning electron microscope, provide constant contrast in the recrystallised grains and mottled contrast (due to stored deformation) in unrecrystallised grains.

Orientation contrast image (OCI) in partially recrystallised Nimonic 901.

Acknowledgement

This research was supported by the DTI *Materials Measurement* programme.

Reference

1. Partially Recrystallised Nimonic Microstructures, *UK Interlaboratory Comparison*; NPL Report, CMMT(A)5, October 1995; B Roebuck, M Stewart, R K Varma and L P Orkney.

For further information contact:

Dr B Roebuck
Centre for Materials Measurement and Technology
Tel: 020 8943 6298
Fax: 020 8943 2989
Email: Bryan.Roebuck@npl.co.uk

Experts

[Link to Expert](#)

National Physical Laboratory
Queens Road
Teddington
Middlesex
United Kingdom
TW11 0LW

Tel: 020 8977 3222
Fax: 020 8943 6458
Email: materials@npl.co.uk

CMMT(MN)001

**Uncertainties in the Measurement of Partially Recrystallised Microstructures
October 1996**

© Crown Copyright 1996. Reproduced by permission of the Controller of HMSO.