



**PARTIALLY RECRYSTALLISED NIMONIC
MICROSTRUCTURES**

A UK Interlaboratory Comparison

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ABSTRACT

A collaborative UK exercise was conducted to investigate the uncertainties associated with the measurement of important microstructural parameters in a partially recrystallised Nimonic 901 alloy. Measurements were made of recrystallised volume fraction, V_R , and grain size of recrystallised, d_R , and unrecrystallised, d_{UR} grains. The measurements were performed on a longitudinal section of an axisymmetric compression testpiece which had been deformed at 1000 °C at a strain rate of 1 s^{-1} . The 95% confidence limits, (two standard deviations), associated with the measurement of V_R , d_R and d_{UR} were respectively $\pm 20\%$, $\pm 40\%$ and $\pm 100\%$. For one of the samples examined the uncertainty was approximately halved to about $\pm 10\%$ (V_R) and $\pm 12\%$ (d_R) for measurements made at laboratories with more experience of examining Nimonic microstructures. It was noted that the identification of unrecrystallised grains is based on arbitrary criteria and that there is scope for the development of a method which would differentiate more unambiguously between recrystallised and unrecrystallised grains.

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1 INTRODUCTION

There is an industrial need to control hot deformation processes such that the final engineered product contains the correctly desired microstructure. Industry is developing process control algorithms which include mathematical models of the development of key microstructural features, such as grain size. The models, in many cases, contain constitutive equations which relate microstructural parameters to process parameters such as stress, strain rate and temperature. In order to validate and develop these models it is necessary to make appropriate measurements of relevant microstructural parameters, either through interrupted hot deformation tests or in-situ if possible. The utility of the models are assessed through sensitivity analyses of key features in the constitutive equations. Thus, it is necessary to have some knowledge of the uncertainties associated with the measurement of the microstructural features of interest.

It is well known that for some standard microstructural measurement methods that the uncertainties are dependent on the procedure adopted, including for example, the number of measurements that are made, the size of the features of interest in relation to the size of the total area investigated and the method of surface preparation. In the current work the microstructure of a partially recrystallised Ni-based alloy (Nimonic 901) was examined through an interlaboratory exercise. No formal standards are directly available for recrystallised and partially recrystallised structures. However, in-house procedures are generally well-established, based on standards for the measurement of grain size or duplex structures, eg ASTM E112 and ASTM E1181 respectively. Some of the issues in the metrology of microstructures have been reviewed by P J Goodhew (Chapter 13 pp 271-289 in "Materials Metrology and Standards" Eds. B F Dyson, M S Loveday and M G Gee, Pub. Chapman and Hall, 1994).

The measurements were made on a longitudinally sectioned axisymmetric testpieces after deformation at 1000 °C to a strain of 0.5 at a strain rate of 1 s^{-1} . One testpiece had been deformed homogeneously. The other testpiece had been subjected to considerable barrelling and the resulting microstructures were not homogeneous across the section. Measurements were made either directly on the polished section or on photocopied micrographs circulated to participating organisations.

The primary objective of the exercise was to examine the uncertainties associated with the measurement of three microstructural parameters.

Volume Fraction Recrystallised

Recrystallised grain size

Unrecrystallised grain size

Seven organisations contributed to the measurements, with about half of the laboratories experienced in making measurements of this kind on Ni-based alloys. The remainder had considerable knowledge of metallography but had developed their skills in the examination of other engineering materials.

2 INTERLABORATORY GUIDELINES

Guidelines were issued to the following participating organisations:

British Steel	Sheffield University
Cambridge University	IMI Titanium
Swansea (IRC) University	MPD Technology
NPL	

Guidelines

Nimonic 901 is the material to be examined, in the form of testpieces from hot axisymmetric compression tests performed at Swansea and Sheffield at 1 s^{-1} strain rate. The test codes are

Source	Code
Swansea	EAT 3
Sheffield	EAT 66

The structures are to be examined at three regions, A, B and C, with the following co-ordinates, where (0,0) is the centre, x and y are horizontal and vertical axes and h and w are the height and width respectively of the compressed testpieces.

A-(0,0); B-(0, 0.95 h/2); C-(0.95w/2, 0)

However, in practice only photocopied micrographs were supplied for position A and this is the only position for which measurements were reported.

The measurements are to be performed on micrographs supplied by NPL and on etched surfaces of the same testpiece circulated to participating organisations.

The participating organisations will measure:

- i) volume fraction recrystallised*
- ii) size of recrystallised and unrecrystallised grains.*

A recommended etch for Nimonic 901 is glyceresia. Other etches are accepted but must be reported.

The method of measurement can be chosen by the participant, ie

*line intercepts
point counting or grids
circle intercepts.*

*At least 200 intercepts, etc should be counted and supplied in tabular form (or on disc as a datafile).
The choice of magnification is open but must be reported.*

3 RELEVANT MICROSTRUCTURAL STANDARDS

Standards of relevance to the intercomparison included:

ASTM E112	Grain Size
	Heyn - Linear
	Jeffries - Circle
ASTM E562	- Volume fraction by Point Counting
ASTM E1181	- Duplex Grain Sizes
ASTM E1382	- Grain size using Image Analysis

Point Counting

The standard for measurement of volume fraction (ASTM E562) recommends the use of a grid where the points on the grid are spaced such that the areas of interest occupy about 0.5 the grid spacing and that about 30 fields of interest are measured to reduce the uncertainty of the measurement. The standard also indicates that photomicrographs are not recommended because of the amount of effort required to obtain an adequate number of fields. To some extent this advice is contradictory in that a round robin in support of the standard is quoted in which photomicrographs were used.

For the current exercise and in other similar measurements on hot compressed testpieces it is unlikely that sufficient area of structure is available at the position of interest to be able to examine a large enough number of fields at the recommended grid spacing. In the present exercise the measurements were made at higher magnifications primarily to enable the unrecrystallised grains to be differentiated from the recrystallised grains but also because of the limited area of material available for investigation. As a consequence the measurements using grids resulted in many grid points falling within each individual area of interest. It may be that this results in an acceptable accuracy, but this would need to be proved by a

round robin on a simulated image where there was no problem differentiating between the two different areas.

Linear intercepts

The linear intercept technique is generally the most convenient method to adopt and this is reflected by the participants in their choice of technique. Several organisations chose to use linear intercepts for volume fraction rather than grids and point counts. The ASTM standard recommends number of intercepts and number of fields required to obtain different levels of uncertainty. The ASTM method of intercept measurement follows a procedure whereby the number of intercepts is counted on a given line length in a field of interest (Heyn). This results in decisions being required on partial intercepts at the end of each line. That is why intercept counting along a circle (Jeffries) is sometimes used since a circle does not have an end.

There is an alternative method of measuring intercepts which avoids the end problem, which is particularly useful for semiautomatic image analysis. The length of line in each grain (along a randomly placed line) is measured to construct a distribution of lengths which can then be analysed. This does not require decisions to be made about line ends. The most important factor is to decide on the number of intercepts to make - usually >200 in a uniform structure.

4 UNCERTAINTIES

The expression of uncertainty is not straightforward and is being examined by a number of groups worldwide; several relevant documents have been issued, for example:

Estimating Uncertainties in Testing	British Measurement and Testing Association	Oct 94
Guide to the Expression of Uncertainty in Measurement	ISO/IEC/IUPAC/BIPM IUPAP/OIML/IFCC	1994
Guide to the Expression of Uncertainties in Testing	NIS 80 NAMAS	Sept 1994
Measurement Uncertainty	ISO TC69 SC6 WG3 (BSI (issued for comment)	Feb 1993)

Some of these documents are working drafts and later issues may now be available. The first document from BMTA is probably the most easily digestible and copies can be obtained from

BMTA, PO Box 101, Teddington, Middlesex TW11 0NQ

It gives simple, straightforward examples of typical estimations of uncertainties.

For the current report what are called Type A uncertainties (conventional statistical analysis of a series of observation) have been used with an expanded uncertainty expressed as twice the standard deviation (ie 95% confidence limits). Type B standard uncertainties from an estimation of errors in the measurement process ie magnification, rulers, software etc have been ignored.

In practice the calculation of standard deviations from less than five measurements is statistically imprecise. However, it is often not cost effective to make sufficient measurements to comply with recommended statistical requirements. The documents listed above discuss the correction factors required for some typical examples where the number of measurements are less than five. The net result is that correction factors are required which are in the range $\times 1.2$ for the 95% confidence limit. A correction factor has been ignored in the current report and the standard deviations have been calculated as though the measurements were obtained from a larger population.

5 MATERIALS AND MICROSTRUCTURE

The Ni base alloy (Nimonic 901) was supplied to NPL as 25 mm diameter rod. This was hot rolled and swaged at NPL (including various anneals between deformations) to 15 mm diameter. The Nimonic 901 was then annealed at 1100 °C for 1 h to further homogenise the structure before subsequent manufacture into compression testpieces. Axisymmetric test cylindrical testpieces either 15 mm long by 10 mm diameter or 10 mm long by 8 mm diameter were machined from the rod for compression testpieces.

After deformation at Swansea (15 x 10 mm) and Sheffield Universities (10 x 8 mm) the testpieces were returned to NPL for examination. Longitudinal sections were prepared and the homogeneous and inhomogeneous structures in the two testpieces can be seen in the low magnification micrographs in Fig 1 which also show the different degree of barrelling. Figs 2 and 3 show intermediate magnification micrographs of EAT 3 (uniform deformation) and EAT 66 (non-uniform deformation) at the centre, side edge and top (bottom) edges of the

longitudinal sections. EAT 3 clearly has a uniform structure across the section whereas, due to extensive barrelling, EAT 66 is considerably inhomogeneous.

Typical higher magnification micrographs of EAT 3 and EAT 66 chosen from the set photocopied and circulated for measurement are shown in Figs 4 and 5. In general the smaller equiaxed grains containing twins are the recrystallised grains. The larger somewhat elongated regions are the unrecrystallised grains.

6 MEASUREMENT METHODS

The measurement methods adopted by the participating organisations included:

Organisation	Volume Fraction	Size
Cambridge	Areas measured by image analysis. Data split arbitrarily at 500 μm^2	
Sheffield - OP1†	Point counts	Circular traverse
Sheffield - OP2†	Point counts	Linear traverse Horizontal - H (longitudinal) Vertical - V (transverse)
MPD	Image Analysis, manually selected areas	Linear intercepts, (ASTM E112). Unrecrystallised structures measured long and transverse
NPL - OP1†	Linear intercepts manual	Linear intercepts Horizontal - H (longitudinal) Vertical - V (transverse)
NPL - OP2†		Linear intercepts Semi-automated Image Analysis
British Steel	Linear intercept ASTM E112-88	Linear intercept ASTM E112-88
IMI	Image analysis, manual edit - remove unrecryst. grains from image	IBAS image analysis Linear intercept and equivalent diameter circle
Swansea	Linear intercept Manual	Linear intercept Manual

† OP1 and OP2 - different observers

7 RESULTS AND DISCUSSION

Results were obtained according to the following table due to limitations of time available.

	Photocopied micrographs	Direct measurements	Experience [†]
Swansea U.	Y	N	R
MPD Technology	Y	Y	R
Sheffield U.	Y	N	R
Cambridge U.	Y	N	L
British Steel	Y	N	L
IMI	Y	N	L
NPL	Y	Y	L

[†] Degree of experience in examination of partially recrystallised Ni-based alloys.
R - reasonable, L - limited

The results of the measurements on the photocopied micrographs and polished samples are given in Tables 1 and 2.

The results are shown graphically in Figs 6-8.

Volume Fraction Recrystallised

It can be seen from Tables 1 and 2 and Fig 6 that MPD Technology reported results from both photocopied micrographs and the polished samples which were in good agreement on both EAT 3 and EAT 66.

The means and standard deviations of the measurements are given in the following table. The results have been assessed combining both micrograph and sample measurements and are separated according to the degree of experience of the operators.

Volume Fraction Recrystallised

Sample	Experience ⁺	Mean Value	1 Standard Deviation (±%) - SD
EAT 3	R	0.634	0.036 (6)
	L	0.652	0.12 (18)
	R+L	0.642	0.074 (12)
EAT 66	R	0.699	0.058 (8)
	L	0.691	0.077 (11)
	R+L	0.696	0.061 (9)

⁺ R - Reasonable, L - Limited; experience of measuring Ni-base alloy microstructures.

It can be seen that there is a significant decrease in uncertainty when the measurements were restricted to the laboratories with more experience. The results indicated that a typical uncertainty of about $\pm 10\%$ (1 SD) should therefore be expected from a comparison of results reported by different organisations, but potentially $\pm 5\%$ (1 SD) might be expected from observers with more experience of measuring similar microstructures. Although different measurement methods were used, ie point counting, linear intercepts and image analysis too few results were available from each technique to comment on the effects of differences in method used. However, for those laboratories with reasonable experience of assessing these microstructures image analysis gave lower results than point counting. Point counting in turn gave lower values than the linear intercept method.

Recrystallised Grain Size

The grain size measurements on the recrystallised grains can be summarised as follows:

Recrystallised Grain Size

mean linear intercept, μm

Sample	Experience ⁺	Mean Value	1 Standard Deviation ($\pm\%$) - SD
EAT 3	R	13.1	0.84 (6)
	L	14.8	2.9 (20)
	R+L	13.8	1.9 (14)
EAT 66	R	8.5	1.6 (19)
	L	10.3	2.3 (22)
	R+L	9.2	2.0 (22)

R - Reasonable, L - Limited; experience of measuring Ni-base alloy microstructures.

In this case the uncertainties were dependent on the material sample. On EAT 66 (inhomogeneous) 1 SD was about $\pm 20\text{--}25\%$, more or less irrespective of the degree of experience of the participating laboratory. But for sample EAT 3 the experienced observers, R, differed by about $\pm 6\%$ (1SD) compared with $\pm 20\%$ for the remainder, L. Two of the three measurements made by the inexperienced observers, L, were very close to the mean of $13.1 \mu\text{m}$ from the experienced observers.

The greater uncertainty for sample EAT 66 probably arises from the smaller average grain size in this material and the consequent difficulty of resolving grain boundaries. A higher magnification for the micrograph might have resulted in reduced uncertainties.

Unrecrystallised Grain Size

The grain size measurements on the unrecrystallised grains can also be summarised as follows:

Unrecrystallised Grain Size
mean liner intercept, μm

Sample	Experience ⁺	Mean Value	1 Standard Deviation ($\pm\%$) - SD
EAT 3	R+L	43.3	16.0 (37)
EAT 66	R+L	34.8	23.7 (68)

⁺ R - Reasonable, L - Limited; experience of measuring Ni-base alloy microstructures.

There were considered to be too few results to separate the R and L categories.

In many cases the unrecrystallised grains had aspect ratios of up to 2 (minor to major axis). This shape difference together with the fewer numbers of grains available for measurement, resulted in more difficulty in obtaining values for the unrecrystallised grain size. Some observers did not try to make the measurement at all, some measured in both transverse (V) and longitudinal (H) directions and some one or the other of H and V. This explains to some extent the large uncertainties, 40-70% (1 SD) noted above. The results reported appear to fall into two groups of either about 40-60 μm or 15-25 μm .

If the results for EAT 3 in the upper size range from Swansea, MPD and NPL are examined (essentially excluding the data from Sheffield) then the uncertainties decrease to about $\pm 20\%$ (1SD).

Summary

Notwithstanding the problems in comparing the results due to the different methods that had been adopted it is clear that there were difficulties in some cases in the identification of the recrystallised grains.

The confidence limits associated with the results overall can thus be summarised as follows:

Parameter	95% Confidence Limits (2SDs)	
	All observers	More experienced observers
Volume fraction recrystallised, V_R	$\sim \pm 20\%$	$\sim \pm 10\%$
Recrystallised grain size, d_R	$\sim \pm 40\%$	$\sim \pm 12\%$
Unrecrystallised grain size, d_{uR}	$\sim \pm 100\%$	$\sim \pm 100\%$

8 ORIENTATION CONTRAST IMAGING

The recognition of unrecrystallised or recrystallised grains in optical images is based on arbitrary criteria. The possibility of using a new technique, Orientation Contrast Imaging (OCI), was briefly examined. The technique uses backscattered electrons to generate contrast differences from grain to grain, resulting from orientation differences between grains. Because unrecrystallised grains contain stored deformation, which in practice consists of many regions within each grain that are misorientated to slightly different degrees, the resulting contrast appears very mottled. However, for recrystallised grains the contrast is uniform across the grain. A typical image from a partially recrystallised Nimonic 901 sample is shown in Fig 9. The image is scratched because of less than optimum specimen preparation but it clearly shows the difference between mottled contrast (unrecrystallised) and constant contrast (recrystallised) grains. This ability to discriminate is thus based on sound physical principles and could be developed into a method to measure, more accurately, recrystallised volume fractions in those materials which give good contrast using electron backscattering techniques.

ACKNOWLEDGEMENTS

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Table 1

**Partially Recrystallised Microstructures
Photocopied Micrographs**

Organisation	Nimonic 901			
	Sample Number EAT	Volume Fraction %	Size (mean linear intercept), μm except + (μm^2)	
			Recrystallised	Unrecrystallised
Swansea	3	68.8	14.0	54.6
	66	76.2	9.2	62.0
Cambridge	3	59.7	125 ⁺	1280 ⁺
Sheffield				
Operator 1	3	62 $\pm$ 8	13.2	24.7
(Circle)	66	65 $\pm$ 8	6.6	19.2
Operator 2	3-H	62 $\pm$ 8	12.9	25.9
(linear)	3-V	62 $\pm$ 8	11.3	21.0
	66-H	65 $\pm$ 8	8.1	19.2
	66-V	65 $\pm$ 8	7.2	14.5
MPD	3	60.9 $\pm$ 4.4	12.5	-
	66	73.5 $\pm$ 2.1	8.0	-
NPL				
Operator 1	3-H	68.1	13.1	42.1
	66-V	70.1	7.8	16.5
British Steel	3	75.4	18.1	too few grains
	66	76.3	12.3	too few grains
IMI	3	52	13.5 (Dcircle)	Not measured
	3		13.1 (mli)	Not measured
	66	61	10.2 (Dcircle)	Not measured
	66		10.9 (mli)	Not measured

H - horizontal (longitudinal), V - vertical (transverse)

Table 2

**Partially Recrystallised Microstructures
Polished Samples**

Organisation	Nimonic			
	Sample Number EAT	Volume Fraction %	Size, μm	
			Recrystallised	Unrecrystallised
MPD	3	60.7 ± 8.3	13.9	59.1 (50 major axis) (85 minor axis)
	66	74.2 ± 8.7	10.9	-
NPL				
Operator 2	3-H		13.3	56.1
	66-V		7.9	59.3

H - horizontal (longitudinal), V - vertical (transverse)

List of Captions

Longitudinal sections of deformed Nimonic 901 testpieces.

Longitudinal sections of EAT 3 (uniform deformation) at centre, side edge (centre line) and top edge (centre line).

Longitudinal sections of EAT 66 (non-uniform deformation) at centre, side edge (centre line) and bottom edge (centre line).

Fig. 4 Typical micrograph of EAT 3 from the photocopied set circulated for measurement.

Typical micrograph of EAT 66 from the photocopied set circulated for measurement.

Volume fraction recrystallised grains.

Grain size of recrystallised grains.

Fig. 8 Grain size of unrecrystallised grains.

Orientation Contrast Image (OCI) of partially recrystallised Nimonic 901.

x8



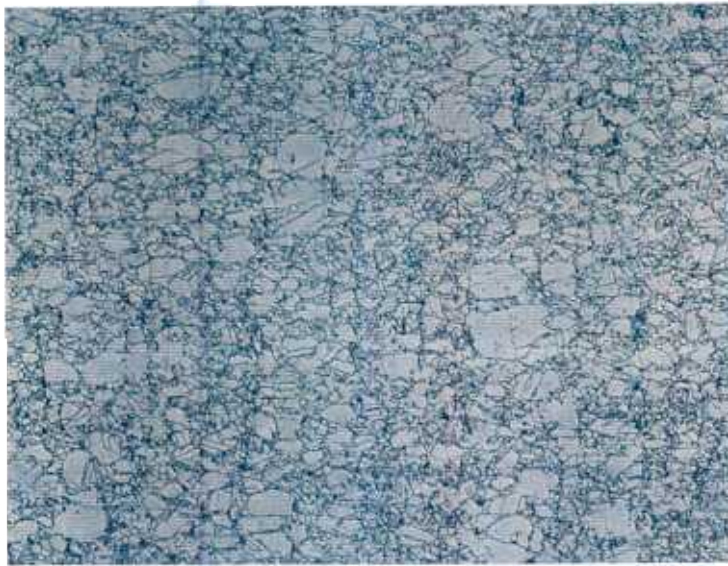
EAT 3

x6

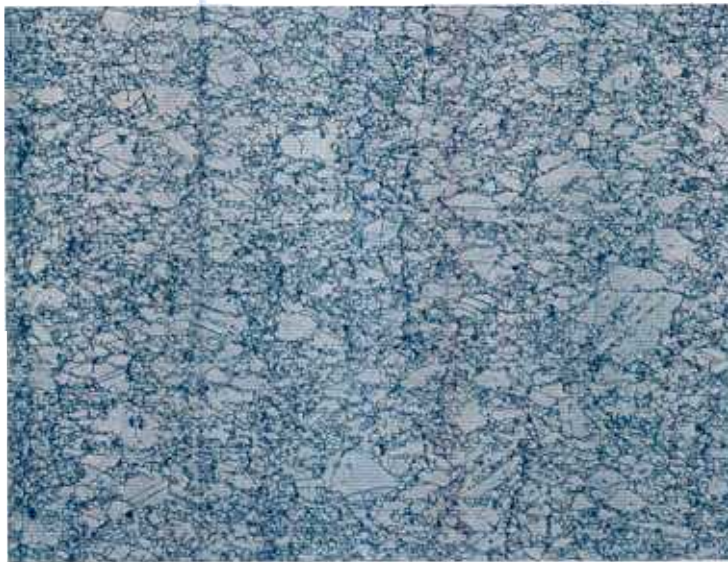


EAT 66

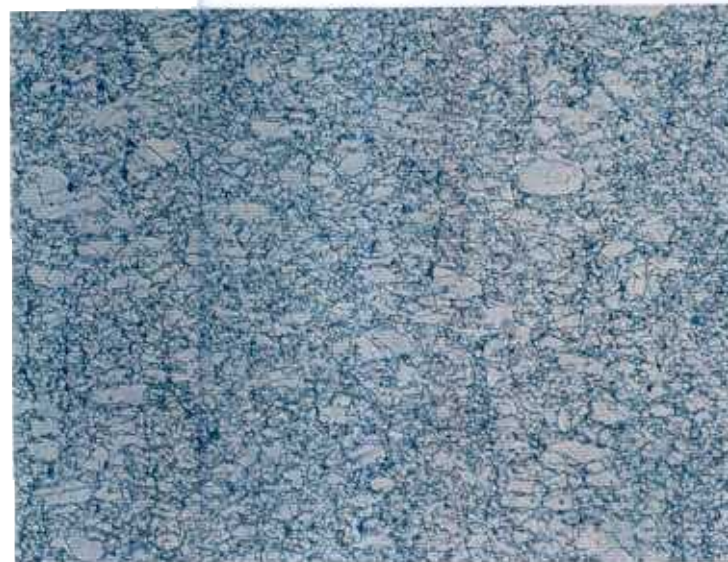
Fig. 1 Longitudinal sections of deformed Nimonic 901 testpieces.



Top edge-Centre line



Side Edge-Centre Line



Centre

EAT 3 x50

Fig. 2 Longitudinal sections of EAT 3 (uniform deformation) at centre, side edge (centre line) and top edge (centre line).



Bottom Edge-Centre Line



Side Edge-Centre Line



Centre

EAT 66 x50

Fig. 3 Longitudinal sections of EAT 66 (non-uniform deformation) at centre, side edge (centre line) and bottom edge (centre line).

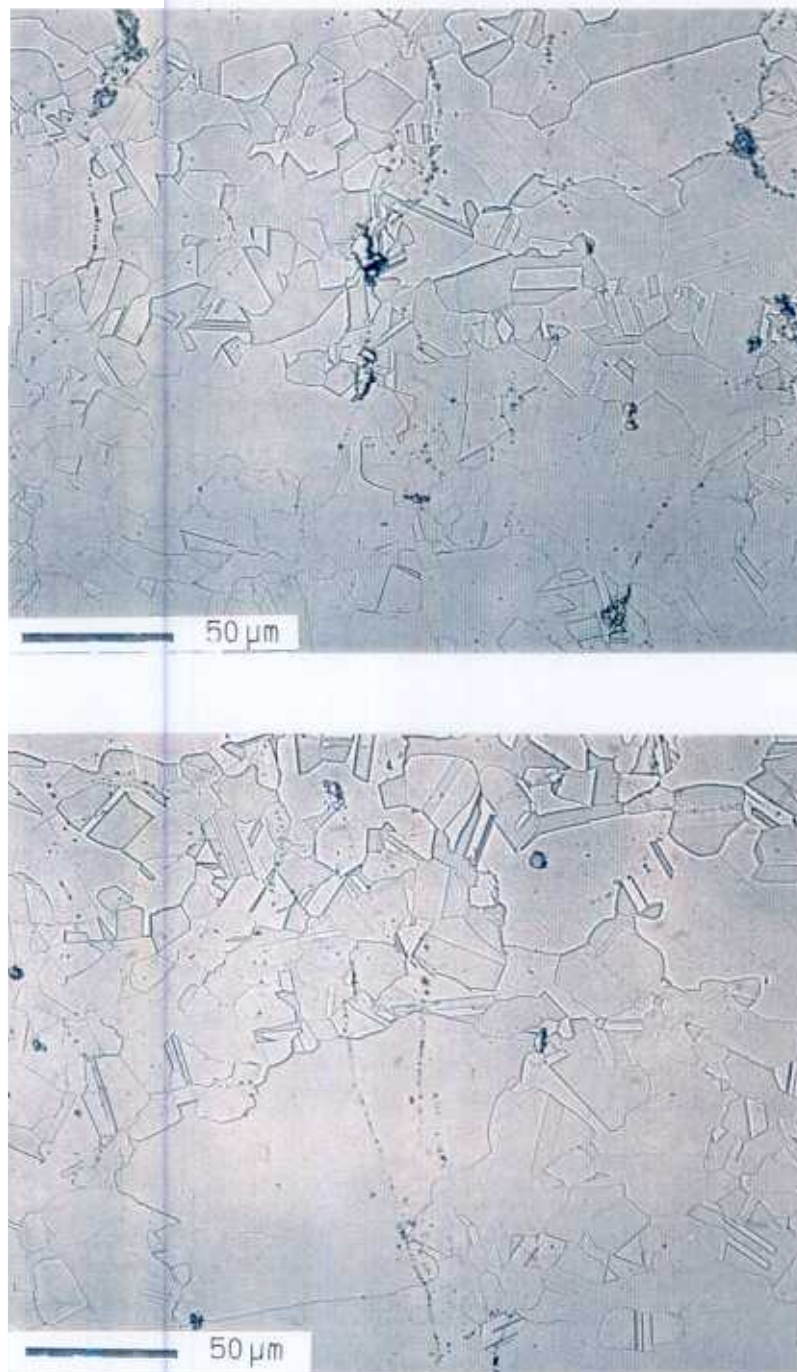


Fig. 4 Typical micrograph of EAT 3 from the photocopied set circulated for measurement.

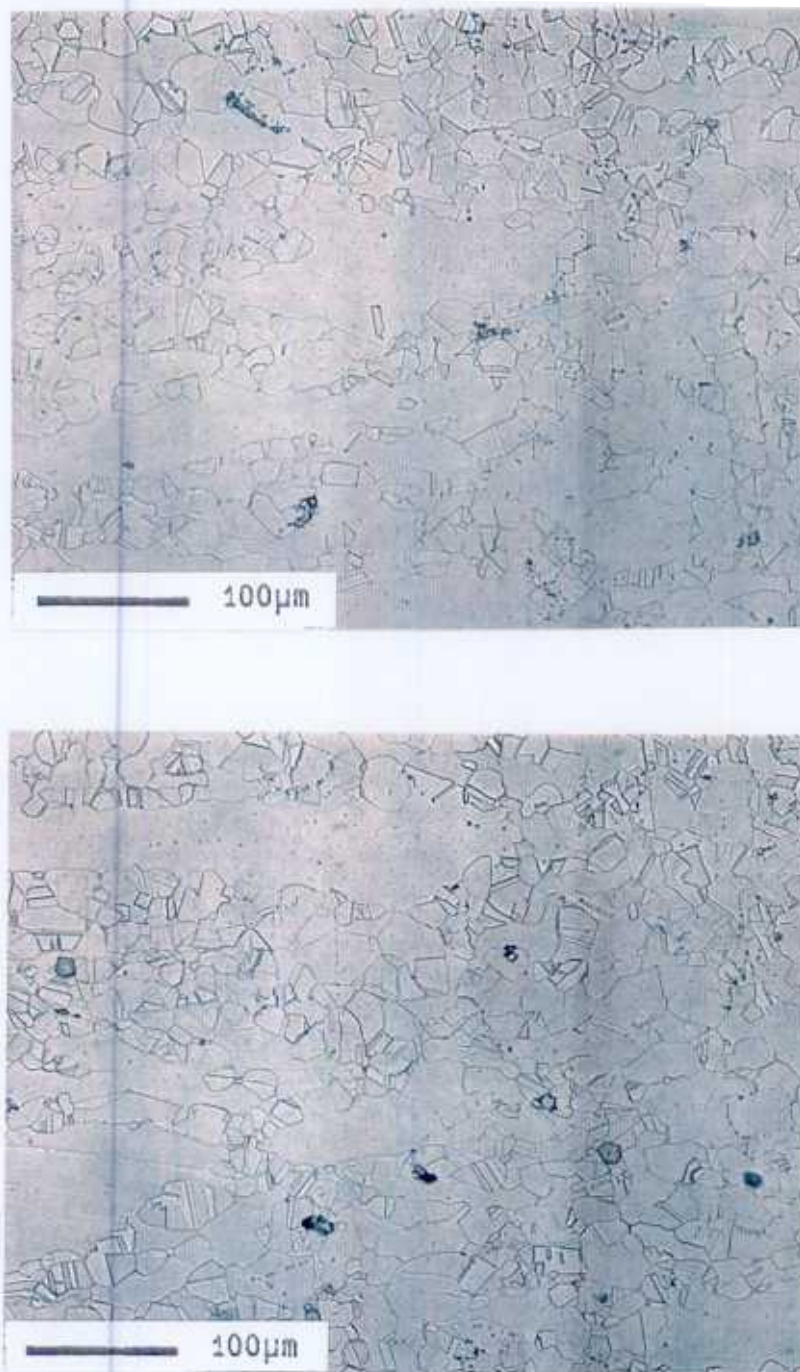


Fig. 5 Typical micrograph of EAT 66 from the photocopied set circulated for measurement.

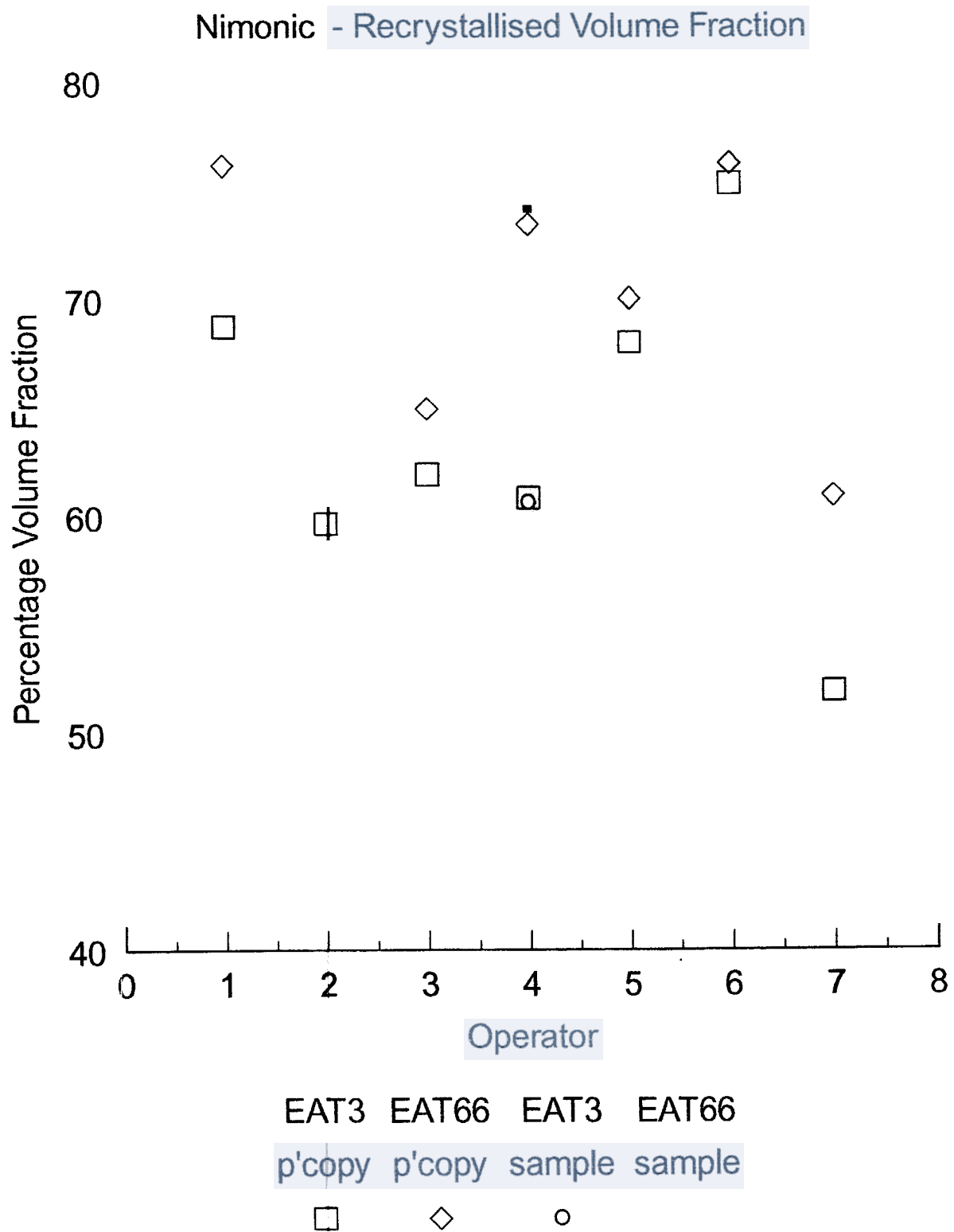


Fig. 6 Volume fraction recrystallised grains.

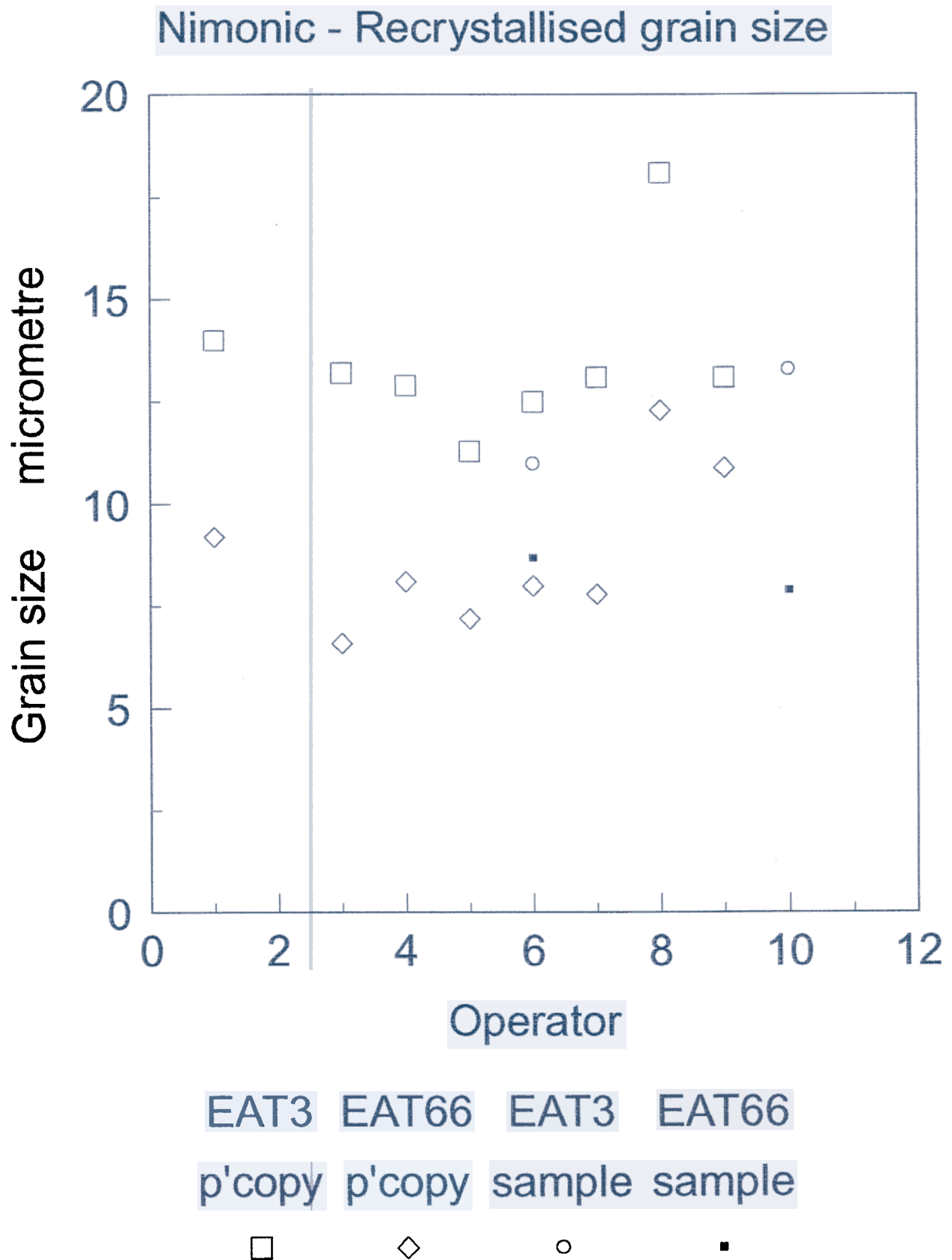


Fig. 7 Grain size of recrystallised grains.

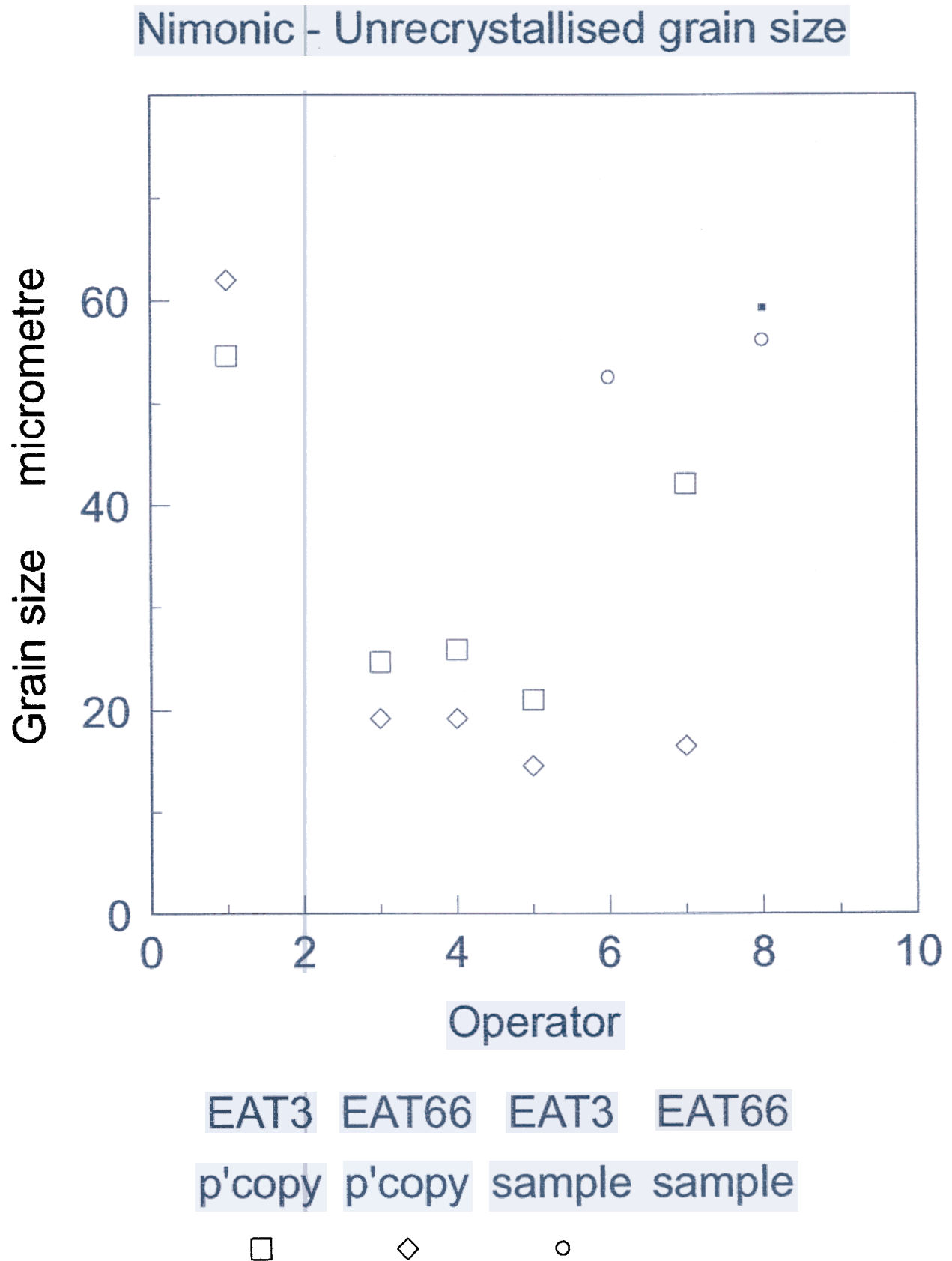


Fig. 8 Grain size of unrecrystallised grains.



Fig. 9 Orientation Contrast Image (OCI) of partially recrystallised Nimonic 901.