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CYCLIC FATIGUE OF HOT-ISOSTATIC-PRESSED SILICON NITRIDE

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ABSTRACT

Cyclic fatigue was studied for hot-isostatic-pressed silicon nitride at room temperature. The number of cycles to failure was 10^6 at a maximum stress of 70 % of the 4-point bending strength and 10^4 cycles at 95 % of the bending strength. The behaviour was found not to be time-dependent but cycle-dependent, at least at the lowest stresses. Scanning electron fractographs revealed numerous small cracks in the fatigued area. These cracks were not observed in other areas of the fractured test bars, nor in samples statically loaded to failure. The findings are consistent with a qualitative model, in which cracks are developed in the glassy phase in the grain boundaries. The cracks grow under the cyclic stress until the sample fails according to the Griffith law. Fracture toughness was measured by the Vickers indentation technique.

Key words: Cyclic fatigue, silicon nitride, hot-isostatic-pressed, four-point bending, fractography, scanning electron microscopy.

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CONTENTS

SUMMARY

Comparatively little is known about the behaviour of ceramics under cyclic loading conditions. It has frequently been suggested that there is no enhanced effect of cycling on crack propagation. Life-time predictions are generally made on the assumption that fracture results from the same phenomenon as from static failure; stress corrosion.

Cyclic fatigue was studied on hot-isostatic-pressed silicon nitride at room temperature. The number of cycles to failure was 10^6 at a maximum stress of 70 % of the breaking strength and 10^4 cycles at 95 % of the flexure strength. The behaviour was found to be cycle-dependent, not time dependent.

Scanning electron fractographs revealed numerous small intergranular cracks in the fatigue zone. The role of cyclic stress is supposed to be an effect of opening and closure of intergranular microcracks and by reactivation of arrested cracks by subsequent cyclic loadings.

1 INTRODUCTION

Ceramic materials have generally been presumed to be insensitive to degradation from cyclic fatigue at room temperature. Some recent reports, however, have given evidence for reduced life-times under cyclic stress for advanced ceramic materials as silicon nitride and zirconia [1-6].

In this work results from cyclic loading of test bars of hot-isostatic-pressed silicon nitride are reported. The fatigue tests were performed in 4-point bending. The fractured surfaces of the samples were investigated by low-power optical microscopy and by high magnification scanning electron microscopy. Fracture toughness was measured with the indentation technique.

2 EXPERIMENTAL

2.1 Material

The test bars consisted of commercial hot-isostatic-pressed silicon nitride, delivered by ABB Cerama, Robertsfors, Sweden. The bars had the dimensions b = 4,5 mm width, h = 3,5 height and length 50 mm. The specimens were cut and prepared by the supplier. The corners on the tensile side were chamfered.

2.2 Test equipment

The four-point test rig was built in accordance with US MIL STANDARD 1942 [7], which is expected to form the basis of the coming ISO Standard for bend testing [8]. Loading span was 20 mm and support span 40 mm. Load was applied through a half-sphere with 45 mm diameter, which was placed in a bowl where the half-sphere could move with very low friction in order to reduce side forces. Both parts were made of hardened steel as the test rig. Cyclic and static loadings were performed in a calibrated MTS servohydraulic testing mechine. The machine is provided with a 40 kN load cell which can be scaled down to 4 kN. All cyclic tests were performed in load control.

To find out the maximum frequency which could be used, a small mirror was glued to one end of the test bars and light from a helium/neon laser was reflected from the mirror to a screen. By increasing the sinusodial frequency at constant stress and register the amplitude of the reflected laserspot on the screen, it was possible to evaluate the frequency dependence of the test equipment. No change in amplitude was observed from 1 Hz up to 40 Hz. This latter frequency was used in the cyclic fatigue tests.

The cyclic loadings were all performed with R = 0,1, where

$$R = \frac{\sigma_{mean} - \sigma_a}{\sigma_{mean} + \sigma_a}$$

σ_{mean} is mean applied stress and σ_a is the amplitude of the applied cyclic stress.

The fracture toughness K_{IC} was measured by Vickers microcrack method using the equation by Evans & Charles (9).

$$K_{IC} = 0,16 \cdot H \cdot a^{1/2} \cdot (c/a)^{-3/2} \qquad (2)$$

where H is hardness, a is half the length of the diagonal in the indentation and c is the crack radius.

Indentation fracture data was obtained with a calibrated Vickers indenter in a Reicherter - Briviskop. The sample was polished to a mirror-like surface with 3 µm diamond-paste. c and a were measured in an optical microscope at 500 X. The sample was tested at loads of 100, 200, 300 and 400 Newton in order to determine the load dependence of the fracture toughness K_{IC}.

2.3 Fractography

Fractured surfaces of broken samples were analyzed with low power microscopy in order to find the general location of the failure origin. The samples were then gold coated by sputtering and examined by scanning electron microscopy (SEM) in a JEOL 35. Most of the work in the SEM was performed at magnifications of 5.500 times.

3 RESULTS

3.1 Cyclic fatigue data

Samples were cycled to failure at three different stress levels σ, where $\sigma = \sigma_{mean} + \sigma_a$; 464, 546 and 625 MPa at 40 Hz.

A number of test bars failed on loading, especially at the highest stress level. According to literature data [10] the 4-point flexural strength of hot-pressed silicon nitride should be 625 - 965 MPa. In a recent IEA-report [11] flexural strength data for 883 ABB-samples obtained by different laboratories in the United States, West Germany and Sweden are reported. The average fracture strength were 650 MPa and the standard deviation 62 MPa. This explains why some of the samples failed on loading.

Most of the samples failed between the two inner rolls. Some failed in the contact point between the test bar and one of the inner rolls. These results have been excluded from the data. The flexural strength data reported contains only samples which failed between the inner rolls at least 2 mm from the contact points. Due to the limited number of test bars, only five results have been obtained for each stress level.

A graphical evaluation of the data from the three stress levels are presented in fig. 1, 2 and 3 in ANNEX. A fit to a log - normal distribution is good even if the standard deviation is high for the highest stress level.

Figure 1 shows the S-N curve obtained for the hot-isostatic-pressed silicon nitride. As can be seen from the figure the samples tested at the lowest stress level on average last for 10^6 cycles while samples at the highest level on average last 10^4 cycles.

The relation between the number of cycles to failure and applied stress, can for a number of materials be written as:

$$\sigma \cdot N^{\alpha} = \text{constant} \qquad (3)$$

A typical value of α is 0,1.
In figure 2 log σ is plotted as a function of log N.
From the slope

$$\frac{d\,(\log \sigma)}{d\,(\log N)}$$

α is found to be 0,07.

3.2 Low frequency and static loads

Since the S-N curve is obtained at constant frequency, it is not possible to separate dynamic and static effects. Three samples were loaded at 5 Hz at the stress level 546 MPa. The mean time to failure at 40 Hz was approximately 40 minutes. If the failure is a pure effect of time, then the time to failure should still be 40 minutes at 5 Hz. On the other hand if the effect is only depending on the number of loading cycles, the time should increase to five hours and the number of cycles should still be 10^5. The scatter between the values obtained at 5 Hz was large, but the mean value for the number of cycles was still close to 10^5.

One sample was loaded at 490 MPa statically. According to the S-N curve this value should correspond to 3,5 hour to failure if the mechanism for failure is an effect of static load. The sample had not broken after 70 hours which is an indication that the observed behaviour is an effect of the dynamic loading.

3.3 Fracture toughness

Ten values for a and c were measured at each load F. The results of the measurements are shown in fig. 3 and 4.

3.4 Fractography

Test bars which failed both under cyclic fatigue and under loading were examined with both low-power optical microscopy and scanning electron microscopy. In nearly all cases the fracture origin was situated near the surface at one of the chamfered corners. Only in one specimen was an internal initation observed. This sample failed on loading at about 225 MPa.

In the Griffith equation

$$\sigma_f = (Z/Y) \cdot K_{IC} \cdot c^{-1/2} \qquad (4)$$

the factor Z/Y is close to 1. [12]. K_{IC} is the fracture toughness, which was measured to be 4,5 $MN \cdot m^{-3/2}$ according to figure 3. c is the flaw size. In the optical microscope c was measured to be 0,3 mm in the actual test bar.

With this value in equation (4) σ_f is calculated to 250 MPa which is close to the measured 225 MPa in view of the uncertainty in K_{IC}.

In most samples a flat semicircular area extending from the corner could be identified, especially in the samples which had been cycled. In the optical microscope the area looked mirror-like in some samples. Figure 5 shows high-magnification photographs from a sample which was cycled to failure at 625 MPa. The number of cycles were $9{,}4 \cdot 10^4$. Fig. (a) is taken close to the corner inside the semicircular area. Fig. (b) is taken in the middle of the bar, far from the semicircular area. Fig. 6 (a) and (b) show corresponding photographs from a specimen which was cycled to failure at 464 MPa. The number of cycles to failure was $4{,}9 \cdot 10^5$. The pictures shown are typical for all the fatigued samples which were investigated in the SEM.

Inside the semicircular area numerous small cracks could be seen. In the regions of subcritical crack growth where the surface appears rough, hardly any cracks could be seen.

The fracture morphology inside the semicircular area was basically intergranular, even if some transgranular cracks also could be seen. Both the crack width and crack length varied considerably as can be seen from the micrographs. Typical values were 0,1 µm up to 1 µm for the width and 10 to 20 µm for the length. In some cases cracks could be seen running as close as 5 µm.

4 DISCUSSION

Cyclic fatigue data for silicon nitride have recently been reported by several authors.

T. Soma et al [2] investigated sintered silicon nitride from 0,03 Hz up to 3 kHz, where they used a resonant bending test rig at the highest frequencies. With this technique a S-N curve was obtained between 10^1 and 10^{10} cycles. The authors noted that the fatigue in silicon nitride appears to depend principally on number of cycles rather than on time, because the fatigue strength at various frequencies did not show any systematic variation in the number of cycles to failure.

For the stress levels of 450, 550 and 625 MPa, their S-N curve gives approximately the same number of cycles to failure at the lowest loads but less cycles at the highest load.

Kawakubo et al [1] also investigated cyclic fatigue behavoiur of sintered silicon nitride. Their S-N curve gives less cycles to failure than was found in the present report. They used notched specimens however, which could explain the difference.

Nikkilä et al [3] studied sintered and hot-pressed silicon nitride. The S-N curve in the present report falls between the curves for the sintered and hot-pressed material. From their results, the authors concluded that long fatigue lifes can be expected for stress levels lower than 2/3 of the bend strength.

The S-N data in the present investigation do not differ significantly from other published data. It must be stressed however, that the measuring points are few. To get a better statistical reliability, at least ten points should have been obtained at each stress level. A number of measurements should also have been done in order to find a possible cyclic fatigue limit.

Time-dependent fracture under static load occurs at room temperature for silicate glasses and for many other ceramics [13]. Hot-isostatic-pressed silicon nitride contains a thin glass layer in the grain boundaries. Since it is not possible to distinguish between effects from time and number of cycles at tests run at constant frequency, it can be argued that the observed S-N curve is an effect of time [14].

The test which was run during 70 hours at constant load without failure is a clear indication that - at least at this stress level - the number of cycles is the dominant reason for failure. It is quite possible that time could be the dominant reason at other stress levels. A natural investigation should be to load samples statically to failure at different load levels.

From the fractographic studies of cyclic fatigue specimens it is suggested that the role of cyclic stress is to break up grain boundaries. This process takes place between a number of grains and a number of small cracks is created. The cracks are arrested at triplets or other grains and can then be reactivated by subsequent cyclic loadings. This qualitative model is supported by the fact that these small numerous cracks could not be seen in other areas of the sample than in the fatigue area. They were not seen at all in samples which failed from static load.

Cyclic fatigue properties should according to the model depend very much on the properties of the glassy phase in the grain boundaries and the grain size. In order to establish a quantitative model for cyclic fatigue of this material, much more experimental work is needed. Static and cyclic loadings at different loads should be performed also at elevated temperatures and if possible, the properties of the grain boundary phase should be changed.

5 CONCLUSIONS

It has been demonstrated that cyclic fatigue may cause damage to silicon nitride at room temperature.

Scanning electron fractographs of fatigued samples show numerous small cracks in the fatigued zone.

The mechanism for fatigue failure is dependent on the number of cycles and not time dependent, at least not at stresses at about 75 % of the flexure breaking strength.

The role of cyclic stress is supposed to be opening and closing of intergranular microcracks and by reactivation of arrested cracks by going around or across the crystals by subsequent cyclic loadings.

6 ACKNOWLEDGMENTS

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8 FIGURES

Fig 1 S-N curve for hot-isostatic-pressed silicon nitride.
$R = 0,1$. $\sigma_{max} = \sigma_{mean} + \sigma_a$. 40 Hz sinusodial

Fig 2 Log σ as a function of log N for determination of the coefficient α.

Fig 3 Fracture toughness K_{IC} determined by indentation technique as a function of applied load F.

Fig 4 Vickers hardness as a function of applied load F.

Fig 5 Scanning electron fractographs of fracture surfaces of specimen cycled to failure at 625 MPa. Number of cycles was $9,4 \cdot 10^4$. (a) close to failure origin at corner. (b) from the interior of specimen outside the fatigue zone.

Fig 6 Scanning electron fractographs of fracture surfaces of specimen cycled to failure at 456 MPa. Number of cycles to failure $4,1 \cdot 10^5$.
(a) close to corner in fatigue zone.
(b) interior of specimen.

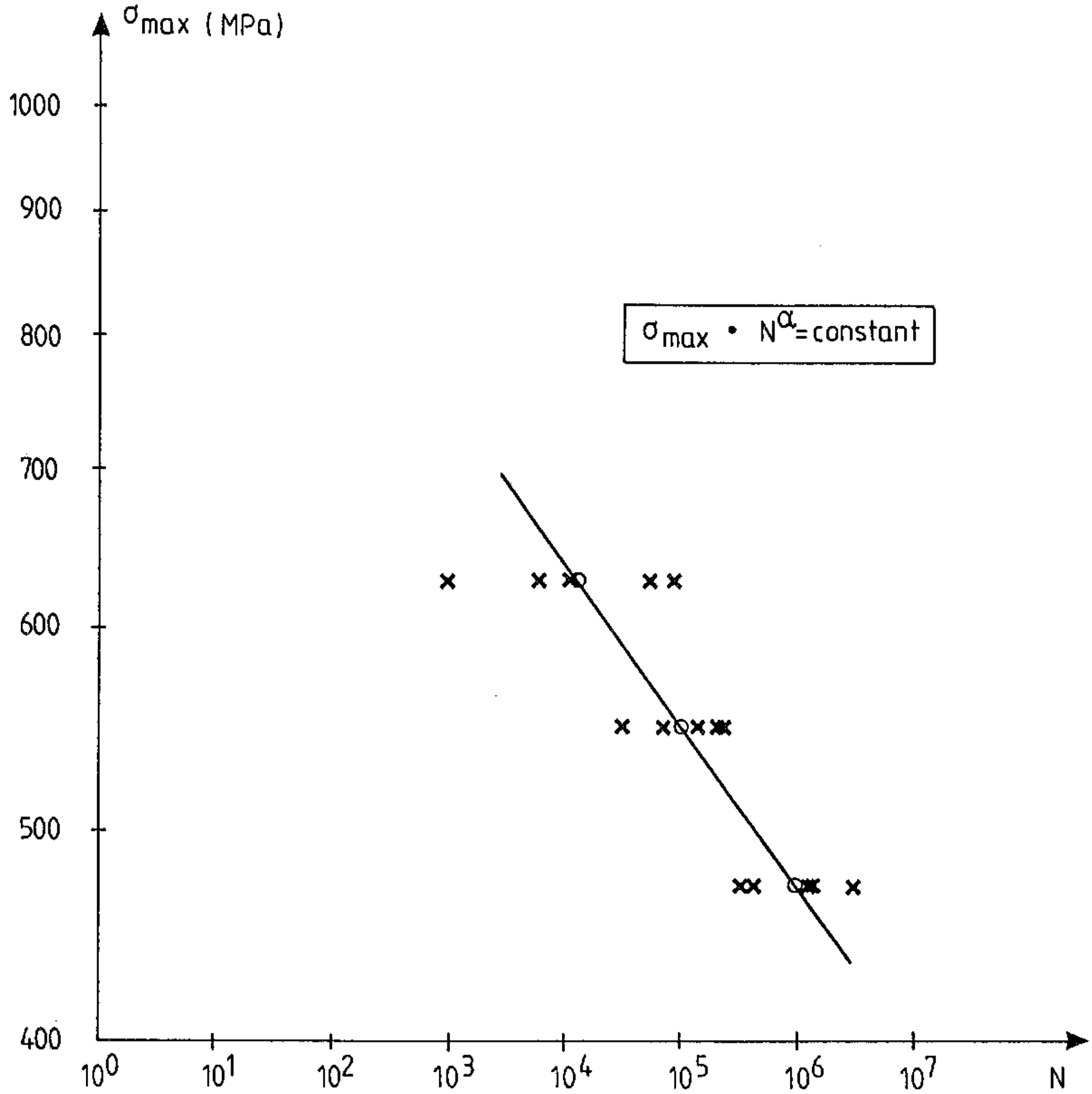


Fig 1 S-N curve for hot-isostatic-pressed silicon nitride.
R = 0,1. $\sigma_{max} = \sigma_{mean} + \sigma_a$. 40 Hz sinusodial

$$\sigma \cdot N^{\alpha} = \text{constant}$$

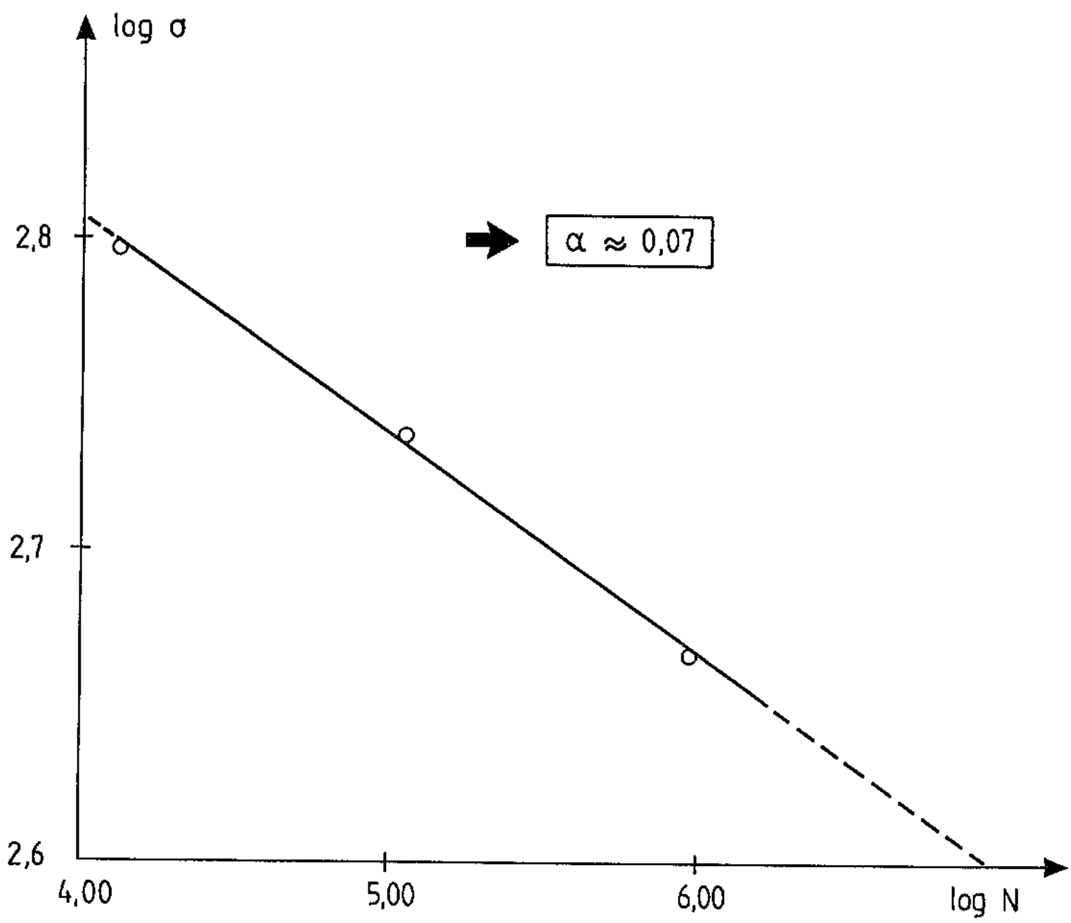


Fig 2   Log σ as a function of log N for determination of the coefficient α.

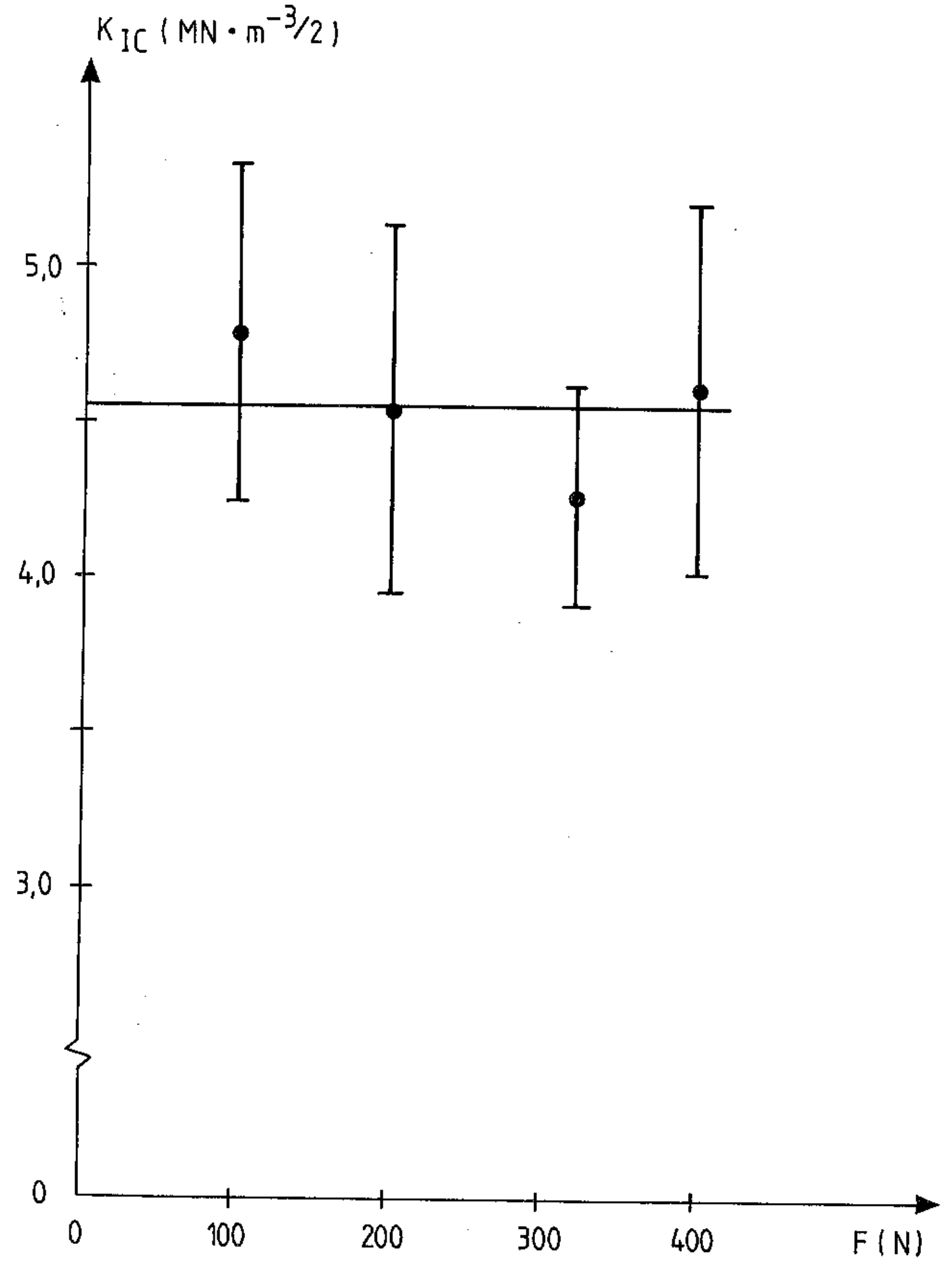


Fig 3 Fracture toughness K_{IC} determined by indentation technique as a function of applied load F.

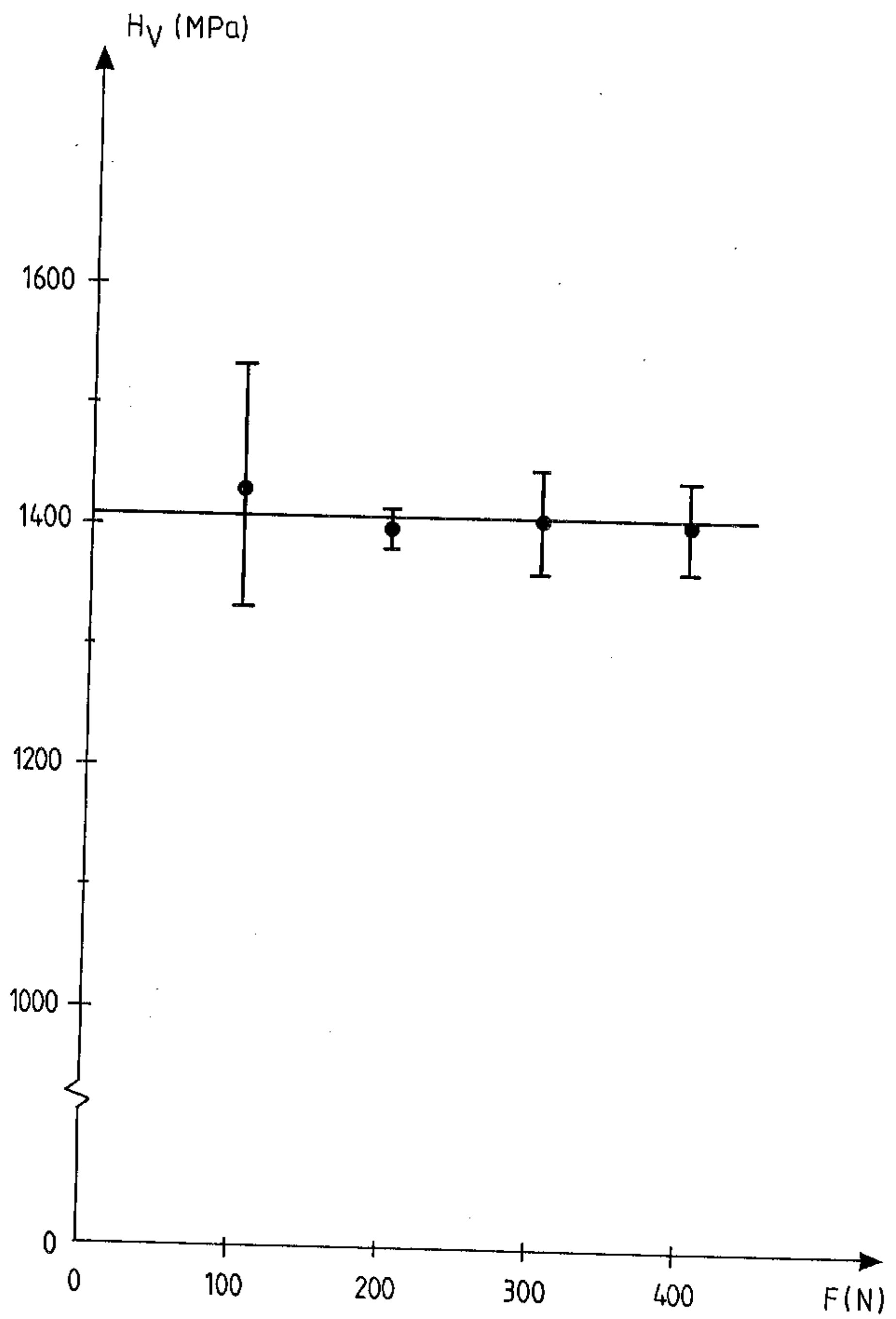
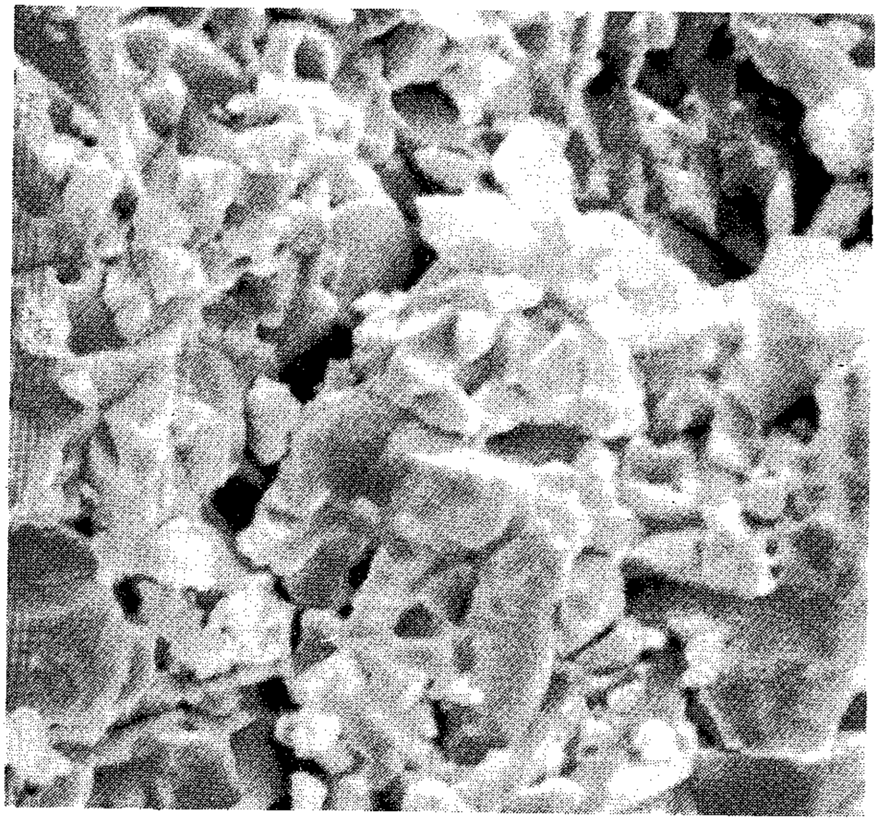
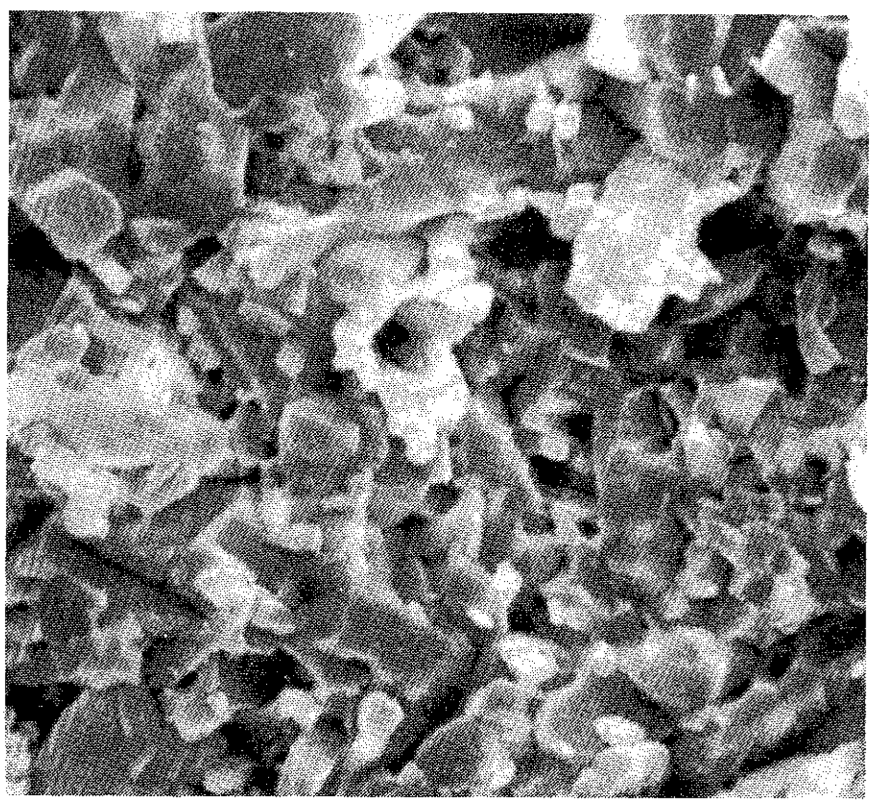


Fig 4 Vickers hardness as a function of applied load F.



(a)

3 μm



(b)

Fig 5 Scanning electron fractographs of fracture surfaces of specimen cycled to failure at 625 MPa. Number of cycles was $9{,}4 \cdot 10^4$. (a) close to failure origin at corner. (b) from the interior of specimen outside the fatigue zone.

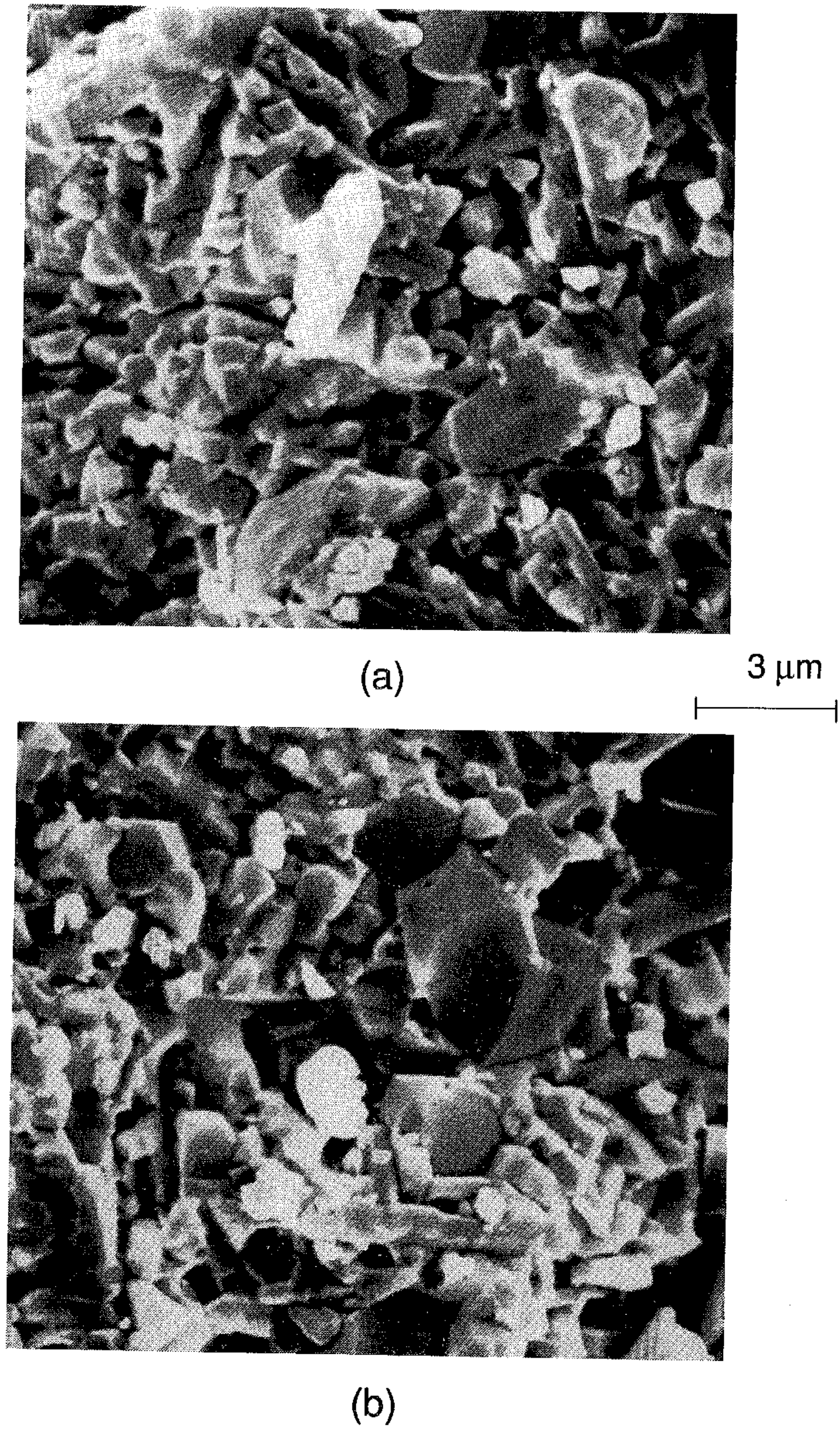
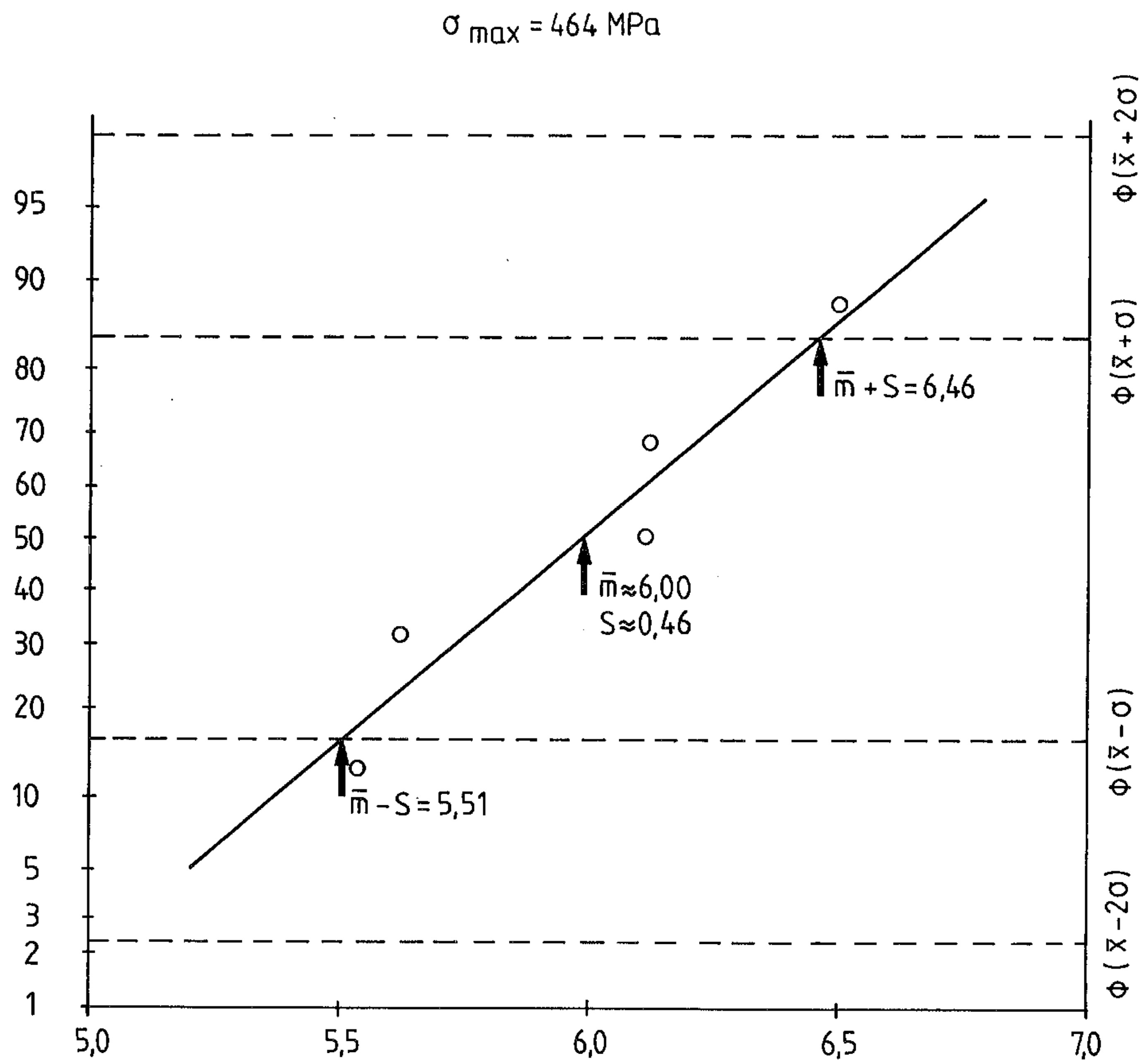


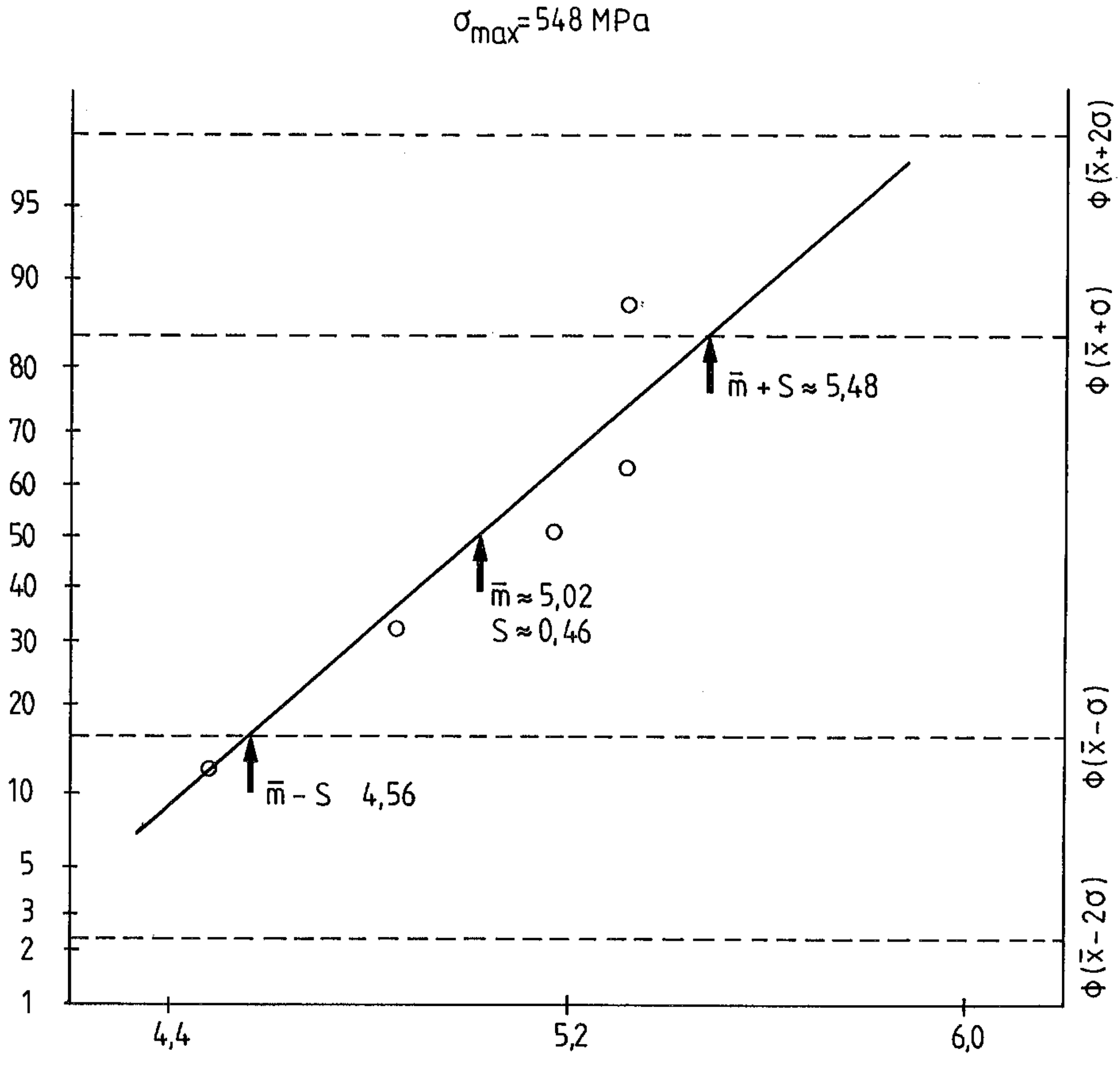
Fig 6 Scanning electron fractographs of fracture surfaces of specimen cycled to failure at 456 MPa. Number of cycles to failure $4,1 \cdot 10^5$.
(a) close to corner in fatigue zone.
(b) interior of specimen.

ANNEX

Grafical evaluation of fatigue data for determination of mean value and standard deviation for the three stress levels.


σ max = 464 MPa
95
90
80
70
60
50
40
30
20
10
5
3
2
1
5,0
5,5
6,0
6,5
7,0
m̄ + S = 6,46
m̄ ≈ 6,00
S ≈ 0,46
m̄ − S = 5,51
Φ(x̄ + 2σ)
Φ(x̄ + σ)
Φ(x̄ − σ)
Φ(x̄ − 2σ)

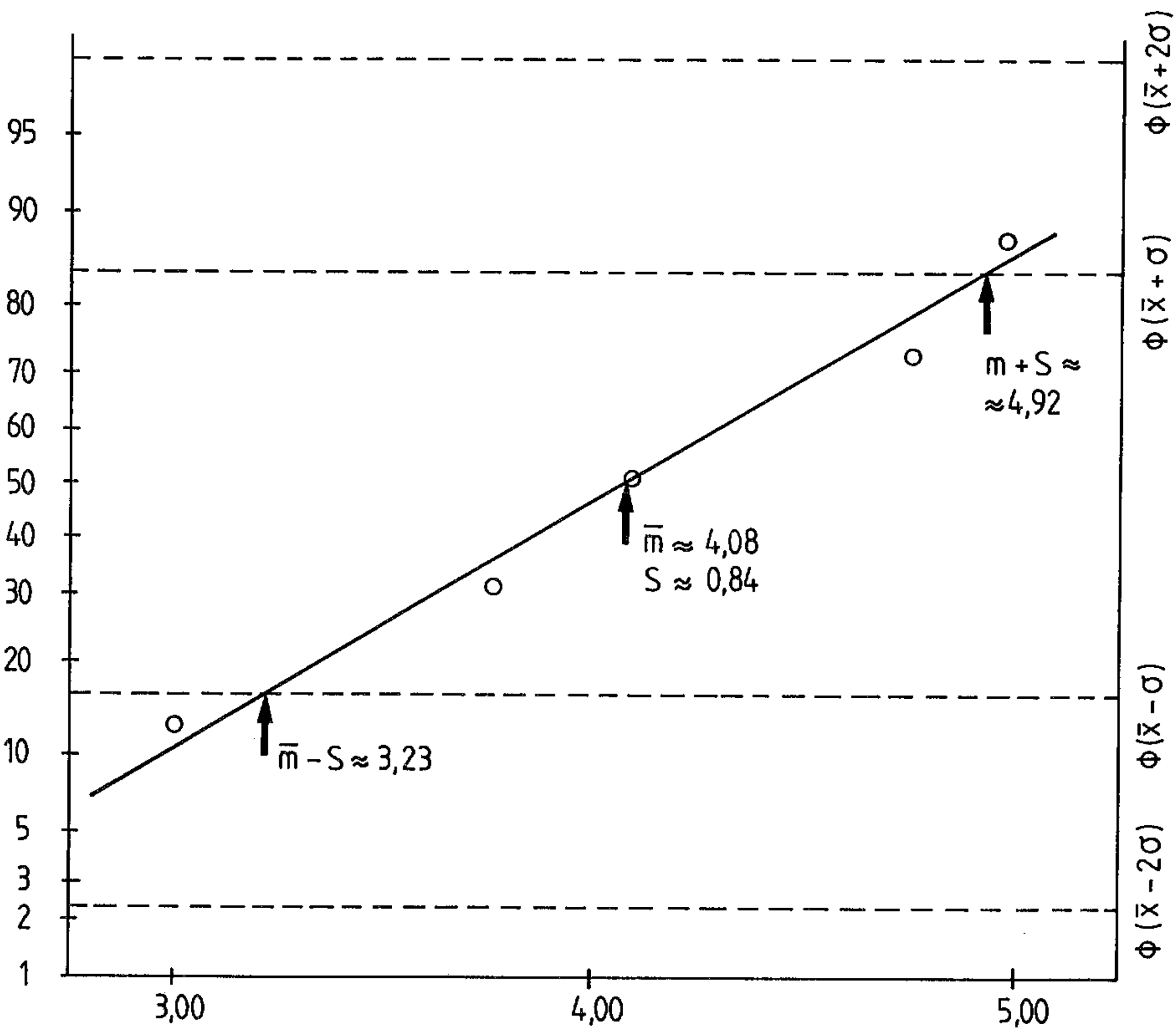
ANNEX Fig 1



σmax= 548 MPa
95
90
80
70
60
50
40
30
20
10
5
3
2
1
4,4
5,2
6,0
m̄ + S ≈ 5,48
m̄ ≈ 5,02
S ≈ 0,46
m̄ − S 4,56
Φ (x̄+2σ)
Φ (x̄+σ)
Φ (x̄−σ)
Φ (x̄−2σ)

ANNEX Fig 2

σ_{max} = 625 MPa



ANNEX Fig 3