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SUBCRITICAL CRACK GROWTH OF WESGO Al-995 ALUMINA AT ROOM TEMPERATURE

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This report contains the results of subcritical crack growth experiments on Wesgo Al-995 alumina.

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CONTENTS

ABSTRACT

For the evaluation of ceramic components subjected to (thermo)-mechanical stresses, it is important to know the subcritical crack growth behaviour of the material. At ECN, research is directed to the development of ceramic components fabricated with strong fine grained ceramics. Subcritical crack growth can restrict the application of ceramics to rather low stress levels if it is not properly designed for.
In the present investigation subcritical crack growth experiments in a 4-point bending device were performed on a commercial alumina Wesgo Al-995. The subcritical crack growth parameters of Wesgo Al-995 alumina are determined with the modified lifetime method.

The crack growth data points show a rather small scatter band around the fit line, which means that the data can be described by a simple power law. Within the experimental conditions there is no indication of a threshold at the lower end of the crack growth curve.

It is recommended to repeat these experiments with a larger series of specimens (at least 20) and at high temperature (1000°C) in order to obtain a better determination of the subcritical crack growth parameters of Wesgo Al-995 alumina.

1. INTRODUCTION

The objective of the IOP-TK project "Advanced Ceramics Testing and Design" is to develop and validate experimental and numerical methods for the failure probability and lifetime prediction of ceramic components. For the evaluation of ceramic components subjected to (thermo-)mechanical stresses, it is important to know the degradation behaviour of the material. The most important failure modes are:

- fast fracture
- subcritical crack growth
- creep
- cyclic fatigue
- corrosion.

Often a combination of these failure modes are involved in the degradation of ceramic components. Subcritical crack growth can cause catastrophic failure within the economic lifetime of a (thermo)-mechanical loaded ceramic component if it is not properly designed for.

Depending on the type of ceramic, subcritical crack growth can occur at low and high temperatures in a non-vacuum environment. Alumina is especially prone to subcritical crack growth in a humid environment. The sensivity of a ceramic material to subcritical crack growth can restrict the application of a ceramic component to rather low stress levels.

To allow correct lifetime predictions the subcritical crack growth data or the power law relation parameters have to be known. Different methods for determining the data are available such as the double torsion method, the double cantilever beam technique, the dynamic bending method and the lifetime method. The first two, which were widely used in the past, are carried out with artificial macroscopic cracks (several millimeters), but in the latter two methods the crack growth of natural cracks are investigated.

It is known that the lifetimes of components cannot be predicted satisfyingly from the data obtained from specimens with large artificial cracks, especially for materials with a strong R-curve effect. It was observed that the growth rate can be more than 2 decades lower

and the exponent of the power law relation by a factor of the order of 4 lower for natural cracks compared with macrocracks of several millimeters [7]. Also the macrocrack methods are usually restricted to relatively high crack growth rates, which are not characteristic for lifetime predictions of components.
The lifetime method offers the possibility to investigate the subcritical crack growth behaviour of the naturally present defects in a ceramic over a large number of magnitudes of the growth rate, even down to a possible threshold. In fig. 1.1 a general type of subcritical crack growth curve is depicted with the crack growth rate versus the stress intensity K.

In creep experiments with Wesgo Al-995 at 1100 and 1200°C it was observed that subcritical crack growth was the dominating failure mechanism [1][2]. In order obtain a better understanding of the subcritical crack growth behaviour of Wesgo Al-995 time-to-fracture experiments were performed in a laboratory environment.

2. EXPERIMENTAL PROCEDURE

2.1. Material

A commercially available alumina (Wesgo Al-995) is used for the investigation. The alumina content is 99.5%, the density is 3.85 kg/dm³ and the mean grain size is 50 µm.

In fig. 2.1 the microstructure of the material is shown, which reveals a lot of pores in the grains and in the grain boundaries. For the preparation of the bending bars, as-sintered bricks were used (300 x 100 x 50 mm). The bricks were cut into sheets, which were subsequently cut into bars of 3.5 x 4.5 x 50 mm. The corners of the bars were chamfered 0.1 mm at 45°.
The four point bending characteristic strength and Weibull modulus of the material are 270 MPa and 21.9 at room temperature. The fracture toughness K_{Ic} is 3.85 MPa$\sqrt{m}$ [4].

2.2. Time-to-fracture measurements

The bending bars were loaded with a dead-weight in four point bending in air. The experimental set-up is shown in figure 2.2. The bending fixture has been made of SiC and was specially designed to minimize the misalignment in the load train. The support rollers of the bending fixture are at a distance of 40 mm and the inner loading span is 20 mm. For the deflection measurement of the specimen between the inner rollers, a special device similar to an arrangement developed by Fett et al [3] was used. The displacement of the specimens were transmitted by a system of three alumina rods on a balance. The deflection in the middle of the inner span was measured with a displacement transducer. With this arrangement only the deflection caused by a constant bending moment is measured, independent of the expansion differences in the system, settlement of the support and roller flattening.
The time-to-fracture was recorded on-line by means of an A/D data-aquisition system.
A detailed description of the apparatus is given in [5], together with an evaluation of the reproducibility and accuracy of the outer fibre bending stress during the experiments.

3. RESULTS AND DISCUSSION

For this investigation 44 time-to-fracture experiments in a laboratory environment (T= ca. 24°C, RH= ca. 40%) were performed. Initially a stress level of 165 MPa was chosen based on a global approximation of subcritical crack growth parameters of alumina. On this stress level 11 tests were run and times-to-fracture upto 115 hours were obtained. At the higher stress levels of 180 and 190 MPa 18 and 15 tests were run respectively. In table 3.1 the details of the time-to-fracture experiments are listed.

The subcritical crack growth of ceramic materials is governed by the stress intensity KI at the crack tip:

$$K_I = \sigma \cdot \sqrt{a} \cdot Y \tag{3.1}$$

with: σ = nominal stress
a = crack length
Y = geometrical factor.

The crack growth rate v

$$v = da/dt \tag{3.2}$$

is usually described by a power law relation of the stress intensity

$$v = A \cdot K_I^n = A^* \cdot (K_I/K_{Ic})^n \tag{3.3}$$

with: A,n = temperature dependent material constants.
K_{Ic} = critical stress intensity or fracture toughness.

For ceramics the exponent n is usually greater than 15.

The time-to-fracture tf is the time until the critical stress intensity K_{Ic} is reached. The time-to-fracture can be determined from (3.1) and (3.3) under the condition of sufficient crack growth

$$t_f = \int_{a_i}^{a_c} \frac{da}{v} = \int_{a_i}^{a_c} \frac{da}{A(\sigma \cdot Y \cdot \sqrt{a})^n} \tag{3.4}$$

Under constant load conditions equation (3.4) can be solved

$$t_f = B \cdot \sigma^{-n} \cdot \sigma_c^{\,n-2} \tag{3.5}$$

with: σ_c = critical stress at the initial crack length

The constants A, B and n can be determined from time-to-fracture test data. For the determination of the subcritical crack growth parameters from the time-to-fracture data the modified lifetime method is used [7].

By combining (3.1) and (3.2) the general equation for the time derivative can be obtained

$$dt = \frac{2}{Y^2 \cdot \sigma^2 \cdot v} K_I dK_I \tag{3.6}$$

Integration from the initial crack length a_i where $K = K_{Ii}$ to the final critical crack length a_c where $K = K_{Ic}$ gives the time-to-fracture

$$t_f = \frac{2}{\sigma^2 \cdot Y^2} \int_{K_{Ii}}^{K_{Ic}} \frac{1}{v} K_I dK_I \tag{3.7}$$

Differentation of equation (3.6) with respect to the initial stress intensity factor and using $K_{Ii}/K_{Ic} = \sigma/\sigma_c$ results in

$$v(K_{Ii}) = - \frac{2\, K_{Ic}^2}{t_f \cdot \sigma_c \cdot Y^2} \; \frac{d[\log(\sigma/\sigma_c)]}{d[\log(t_f \sigma^2)]} \tag{3.8}$$

In the derivation, no special type of crack growth law is prescribed.

In alumina the initial crack size and the initial stress intensity can usually not be identified after a time-to-fracture test. For the determination of the initial stress intensity an indirect procedure is followed, making use of the scatter of the natural cracks and the inherent scatter in the inert bending stress σ_c. These are related by equation (3.1).

In the procedure of evaluating $v(K_{Ii})$ the Weibull data of the bending tests at high stress rates in an inert environment were used [4][6]. The results of the time-to-fracture tests were ranked in ascending order and analysed with the Weibull statistical distribution (see fig. 3.1 to 3.3). The i-th value of the time-to-fracture is associated with the corresponding value of the inert bending stress σ_{ci}, determined with the Weibull data.
For the determination of the derivative part in equation (3.8) the values σ/σ_{ci} and $t_{fi} \cdot \sigma^2$ are plotted in a log-log graph. (see fig. 3.4 to 3.6). The slope of the least squares fit through the data points is equal to the derivative part in (3.8).

For the calculation of the crack growth rate v the geometrical factor Y for natural cracks has to be known. The assumed shape of the crack is a semicircular surface flaw of length a where $Y = 2.24/\sqrt{\pi}$ [8]. The crack growth rates for the individual time-to-fracture tests can now be calculated from (3.8). The results are plotted in fig. 3.7 to 3.9 together with the least squares fit through the data points.
The exponent n and the constant A* can be calculated from the slope and the intercept at $K = K_{Ic}$:

165 Mpa	n = 44	A* = 0.341 m/s
180 MPa	n = 40	A* = 0.105 m/s
190 MPA	n = 24	A* = 0.001 m/s

The crack growth rate data of the three series can be plotted in one figure (fig. 3.10). This figure shows that the data points of the individual series do overlap and that the crack growth rates for a larger K range can be obtained. All the data points can also be combined and analyzed in one fit (see fig. 3.11).

The fit yields an exponent n = 43.5 and a constant A* = 0.4047 m/s. With this result the other subcritical crack growth parameters can be calculated yielding the constant A = 1.5 E-26 and constant B = 1.099 $MPa^2 s$.

Fig. 3.11 shows that the crack growth data points have a rather small scatter band (R squared = 0.92) from the fit line, which means that the data can be well described by the simple power law (3.3). Within the experimental conditions there is no indication of a threshold at the lower end of the curve. Only at the high K range there is some flattening towards a possible plateau in the crack growth curve.

4. CONCLUSIONS AND RECOMMENDATIONS

The subcritical crack growth parameters of Wesgo Al-995 alumina have been determined with the modified lifetime method. The parameters are:

$$v = A^* \cdot (K_I/K_{Ic})^n \qquad A^* = 0.4074 \text{ m/s}, \quad n = 43.5$$

$$t_f = B \cdot \sigma^{-n} \cdot \sigma_c^{n-2} \qquad B = 1.099 \text{ MPa}^2\text{s}, \quad n = 43.5$$

The crack growth data points have a rather small scatter band from the fit line, which means that the data can be described by the simple power law.

Within the experimental conditions there is no indication of a threshold at the lower end of the curve.

It is recommended to repeat these experiments with a larger series of specimens (at least 20) and at high temperature (1000°C) in order to obtain a better determination of the subcritical crack growth parameters of Wesgo Al-995 alumina.

5. ACKNOWLEDGMENTS

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6. REFERENCES

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Table 3.1. List of the time-to-fracture tests.

165 MPa spec.nr.	tf sec.	180 MPa spec.nr.	tf sec.	190 MPa spec.nr	tf sec.
4	33720	29	192	12	18
5	28500	30	19254	13	287
6	41820	31	100	14	46
7	19620	32	663	15	180
8	112920	33	13231	16	18
9	412440	34	336	17	10
27	5100	35	40	18	11
41	31502	36	180	19	60
42	379	37	32	20	16
43	30020	38	778	21	2
44	57	39	834	22	22
		40	647	23	98
		45	84	24	6
		46	3	25	12
		47	302	26	10
		48	705		
		49	13		
		50	2888		

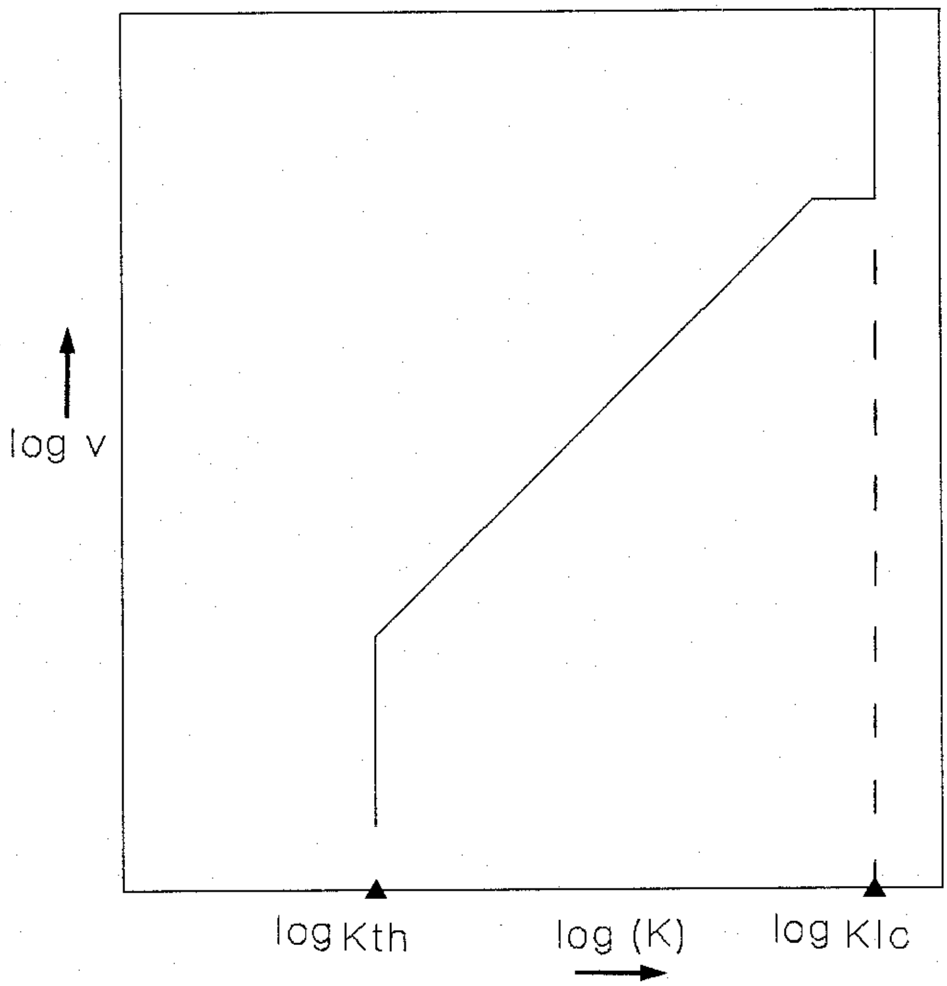

Fig. 1.1. Typical subcritical crack growth curve.

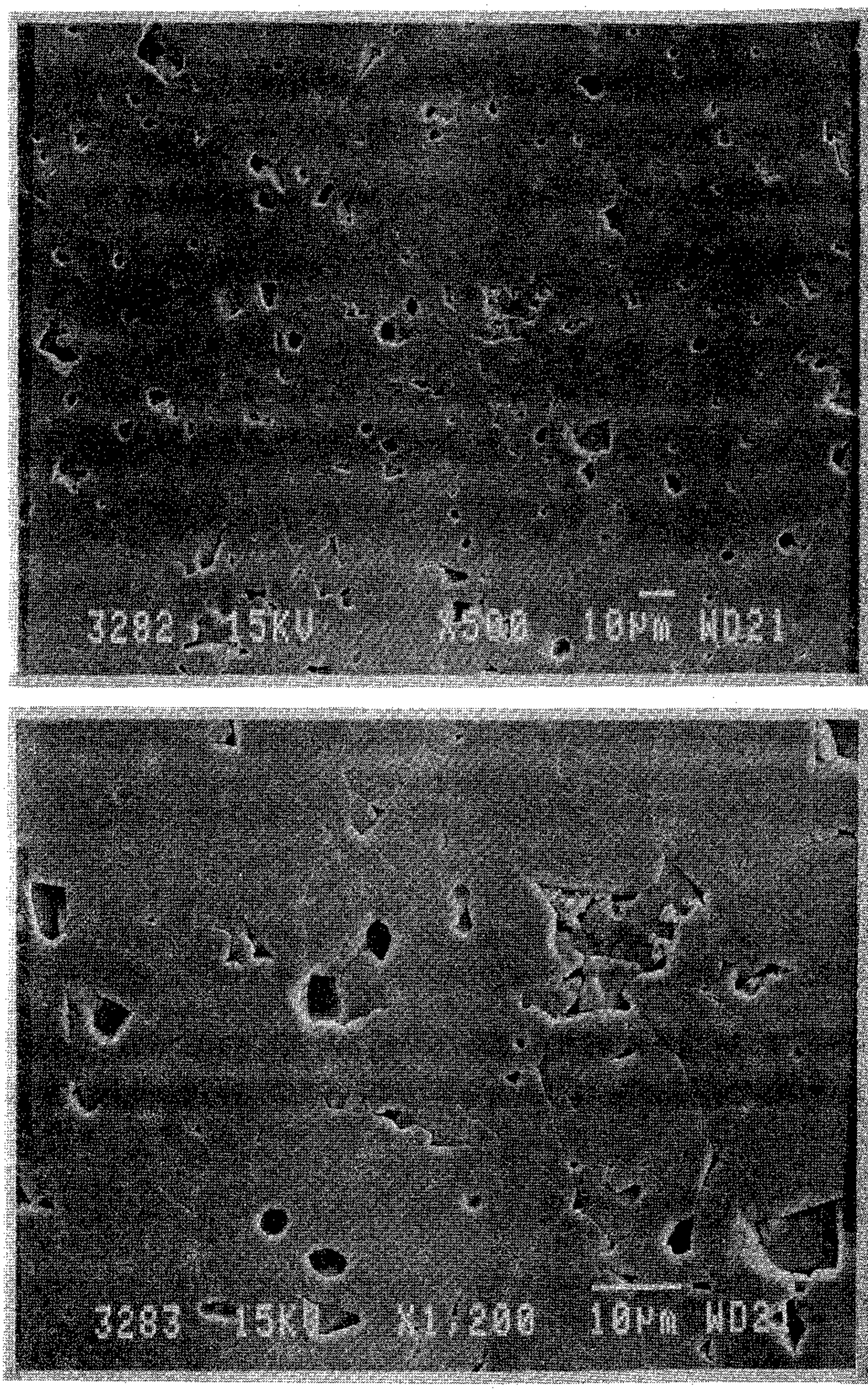

Figure 2.1. The microstructure of Wesgo Al 995.

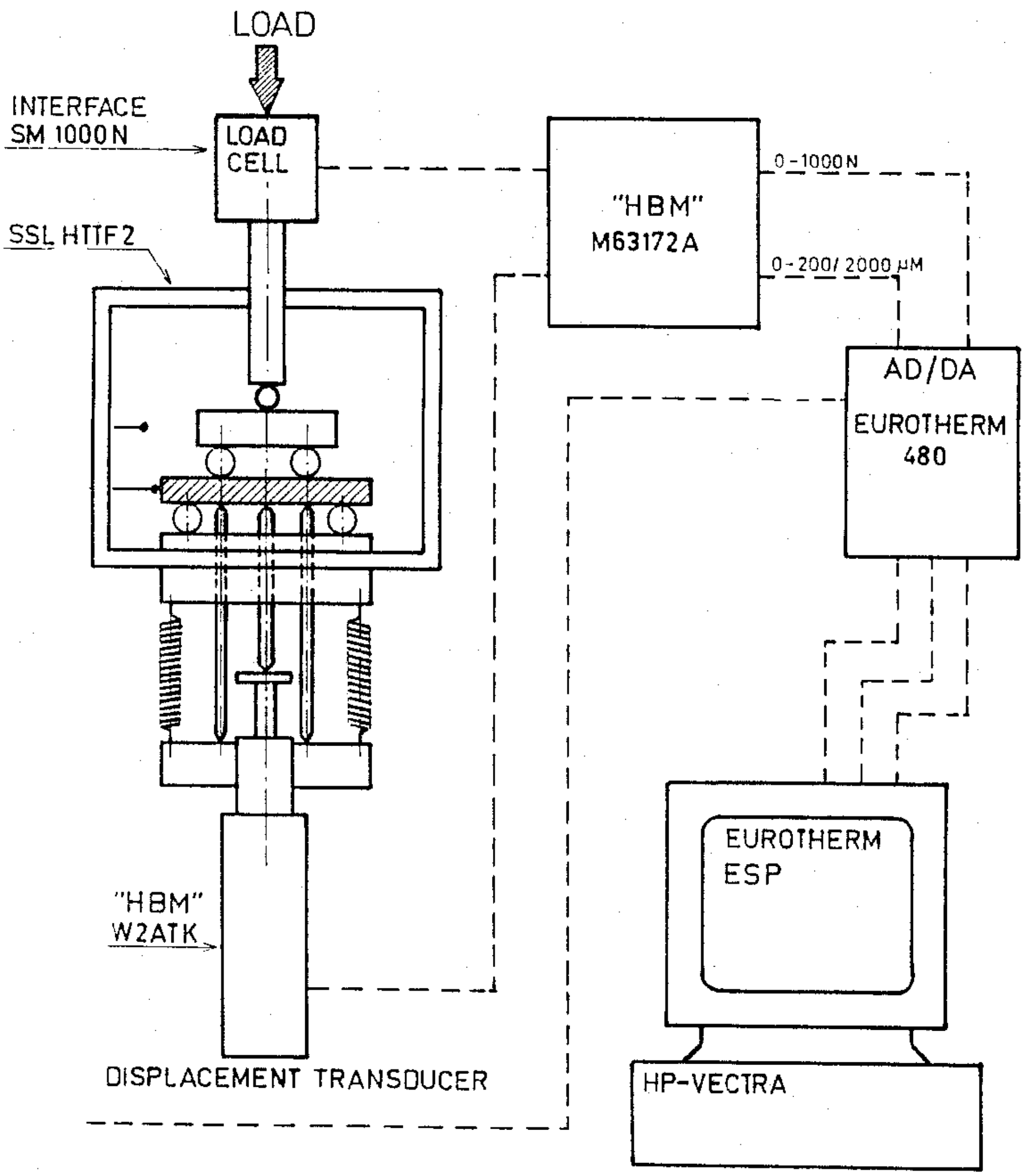

Fig. 2.2. Experimental set-up for the time-to-fracture tests.

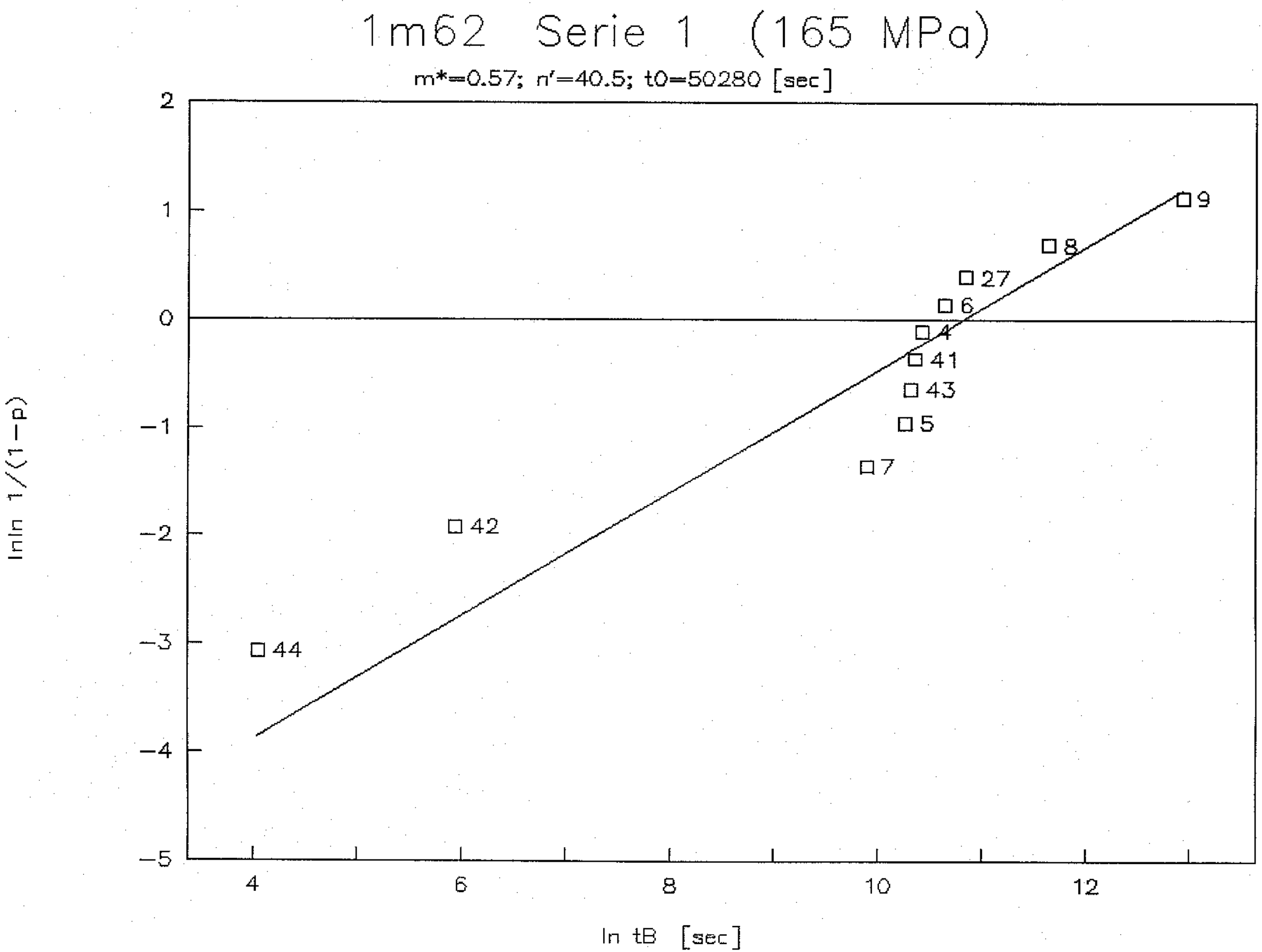

Fig. 3.1 Weibull plot of the time-to-fracture data for σ=165 MPa.

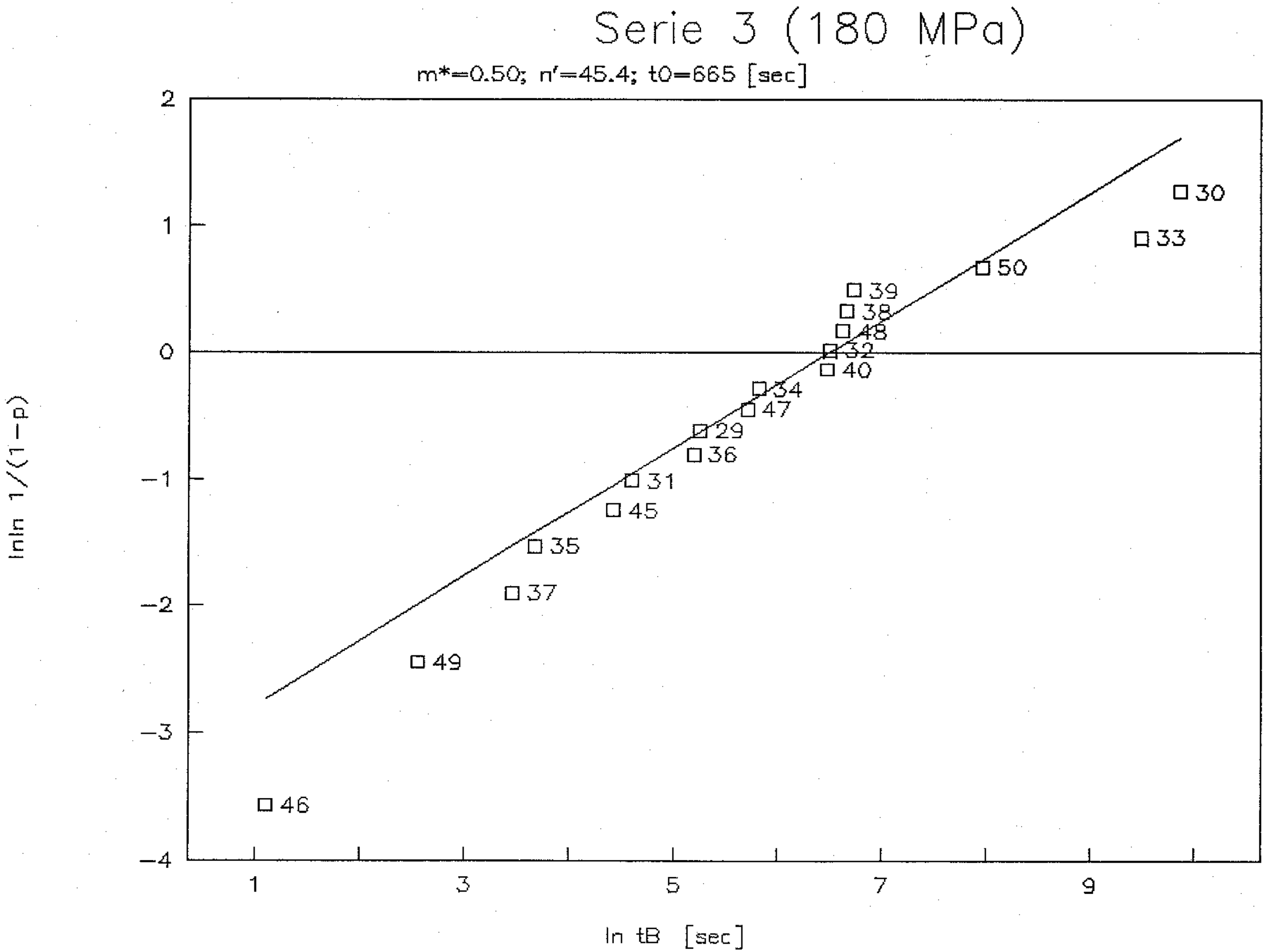

Fig. 3.2. Weibull plot of the time-to-fracture data for σ=180 MPa.

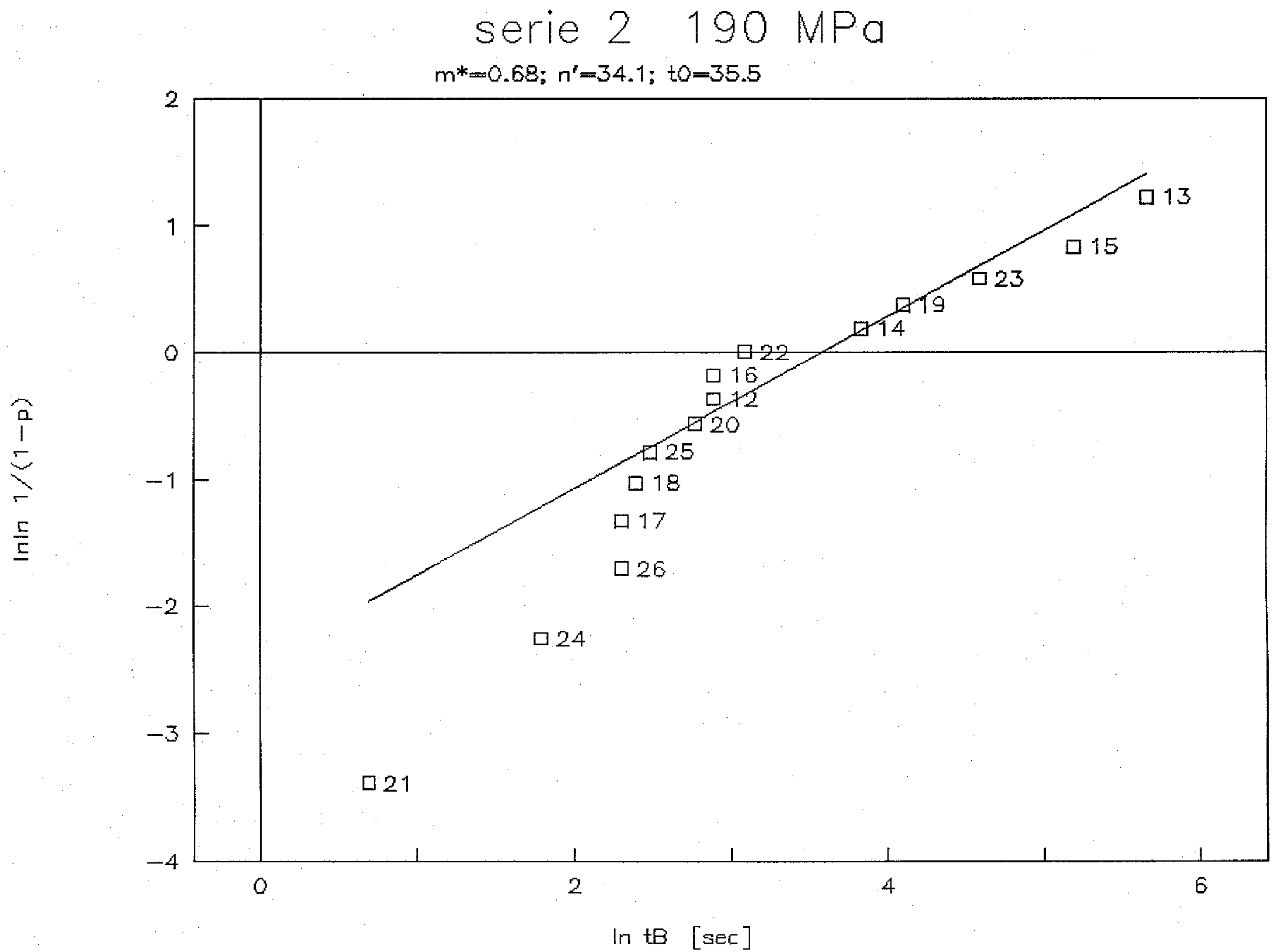

Fig. 3.3. [illegible]ibull plot of the time-to-fracture [illegible]a for σ=190 MPa.

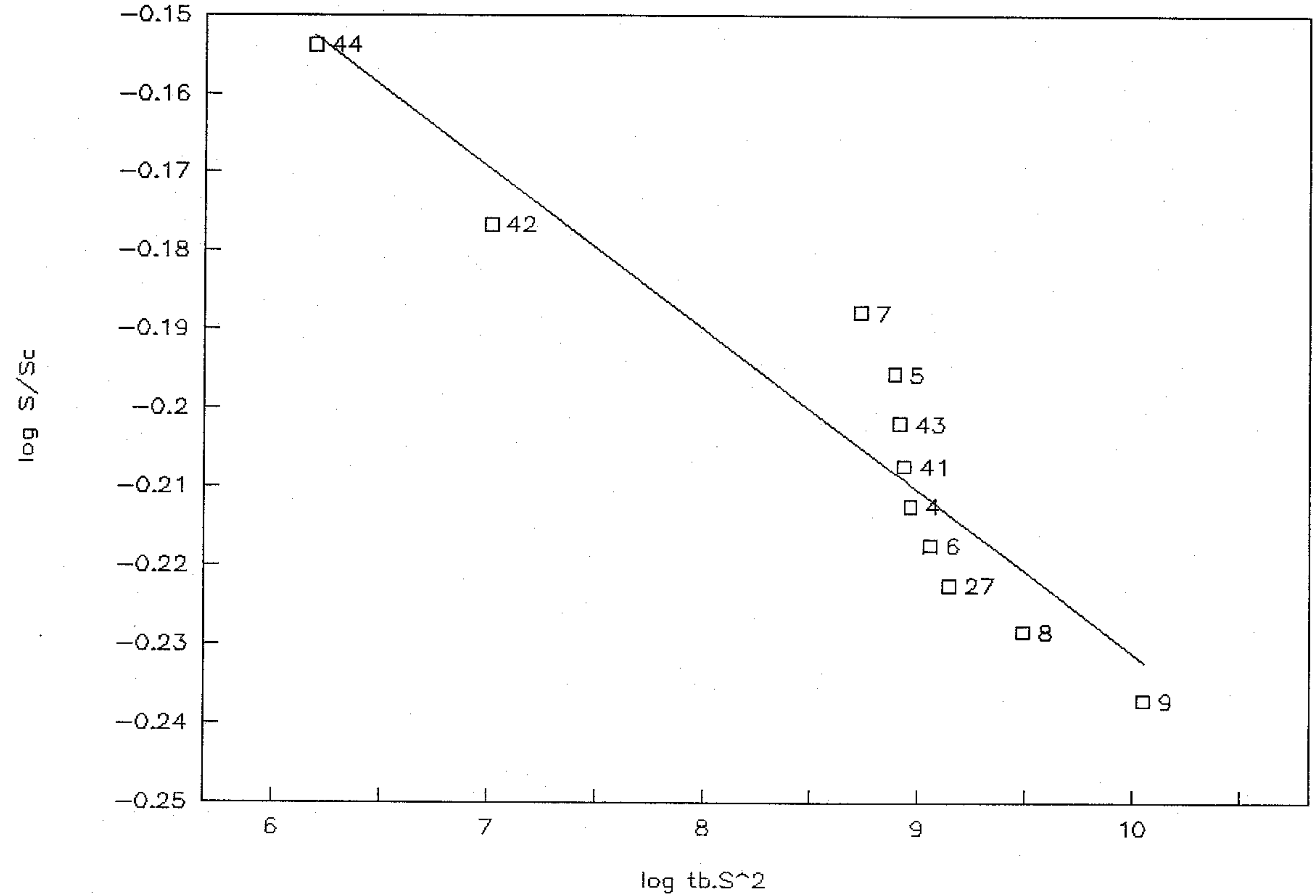

Fig. 3.4. Plot for the determination of the derivative part in the crack growth equation (3.8) for σ=165 MPa.

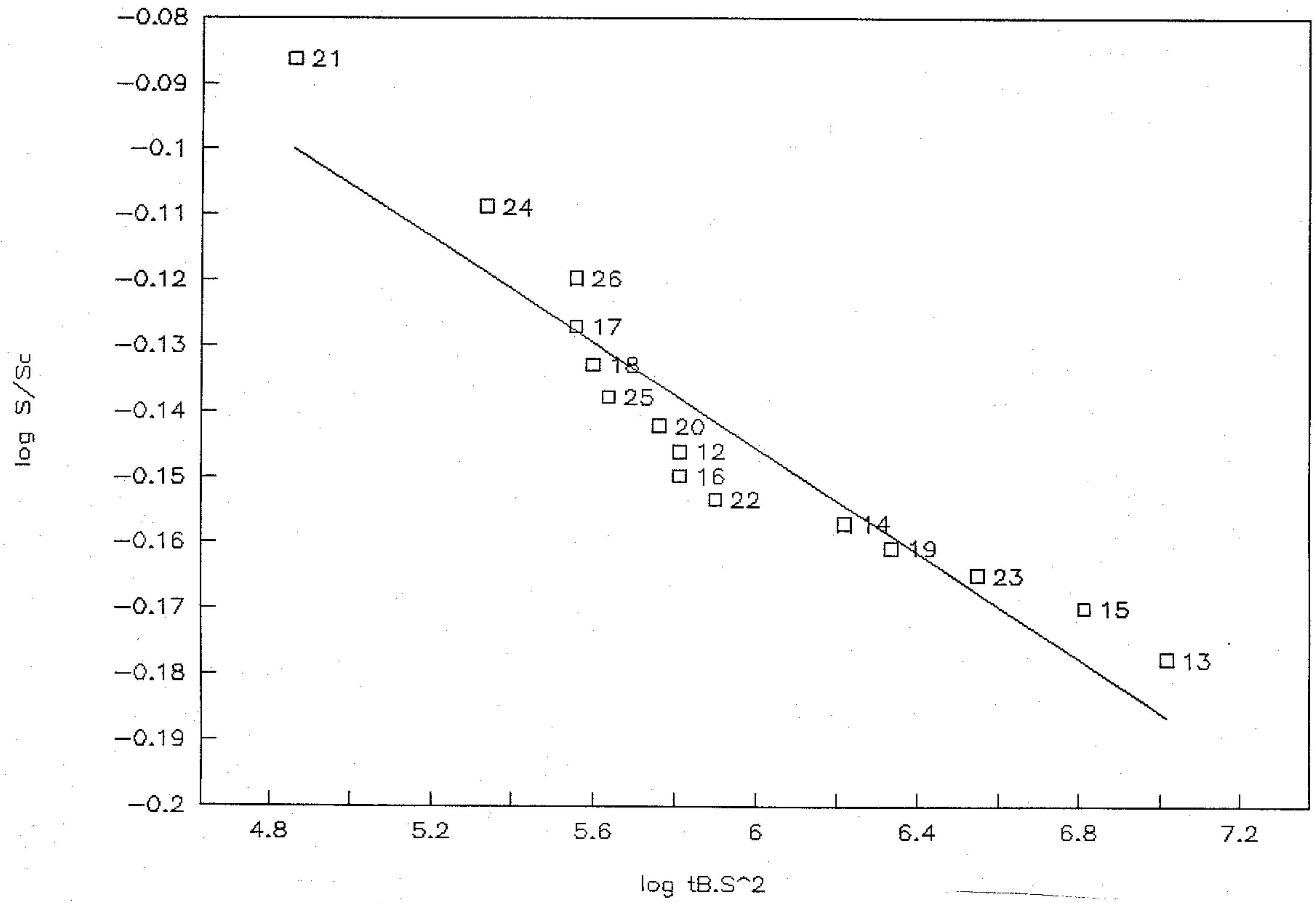

Fig. 3.5. Plot for the determination of the derivative part in the crack growth equation (3.8) for σ=18[illegible]MPa.

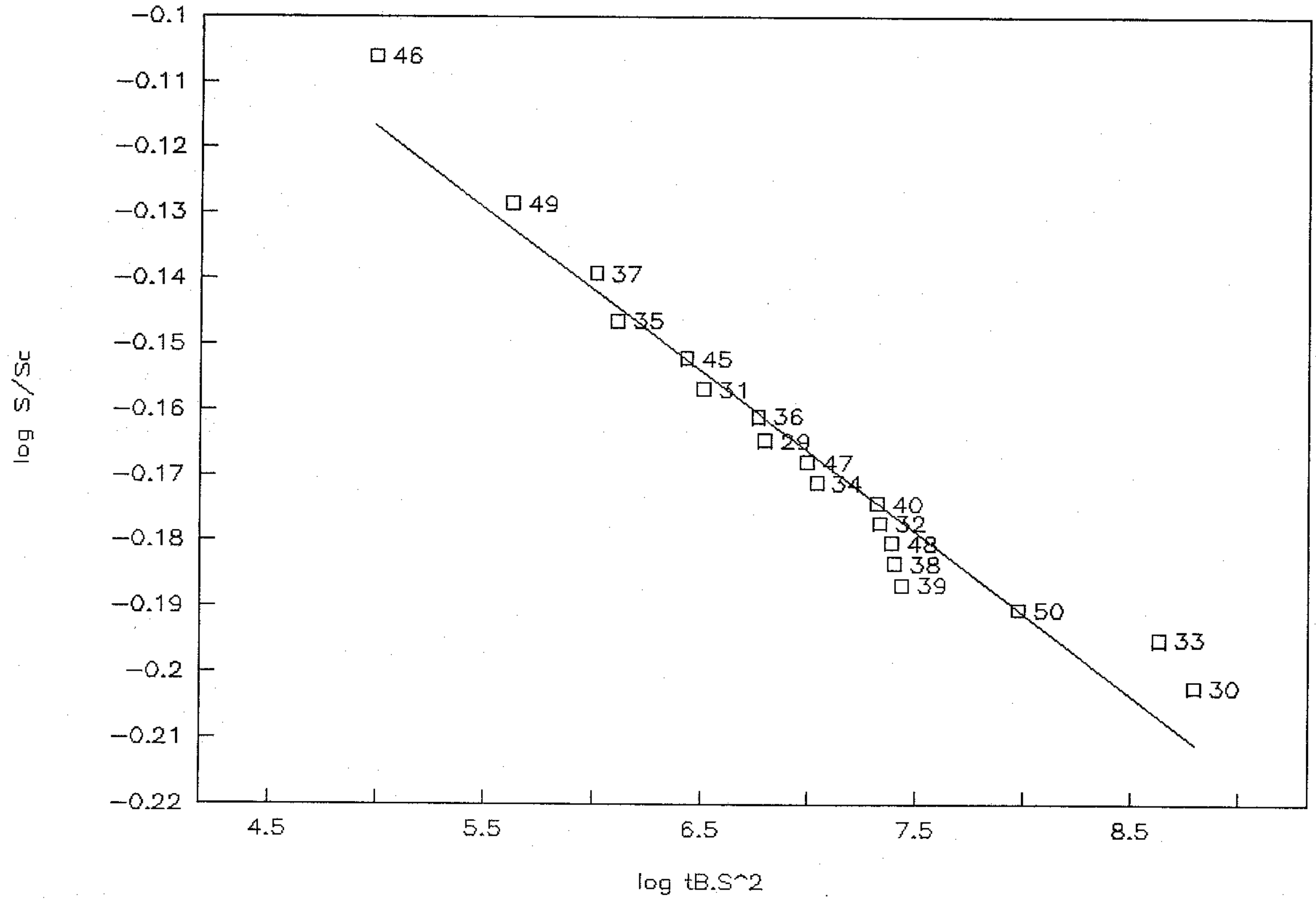

Fig. 3.6. Plot for the determination of the derivative part in the crack growth equation (3.8) for σ=190 MPa.

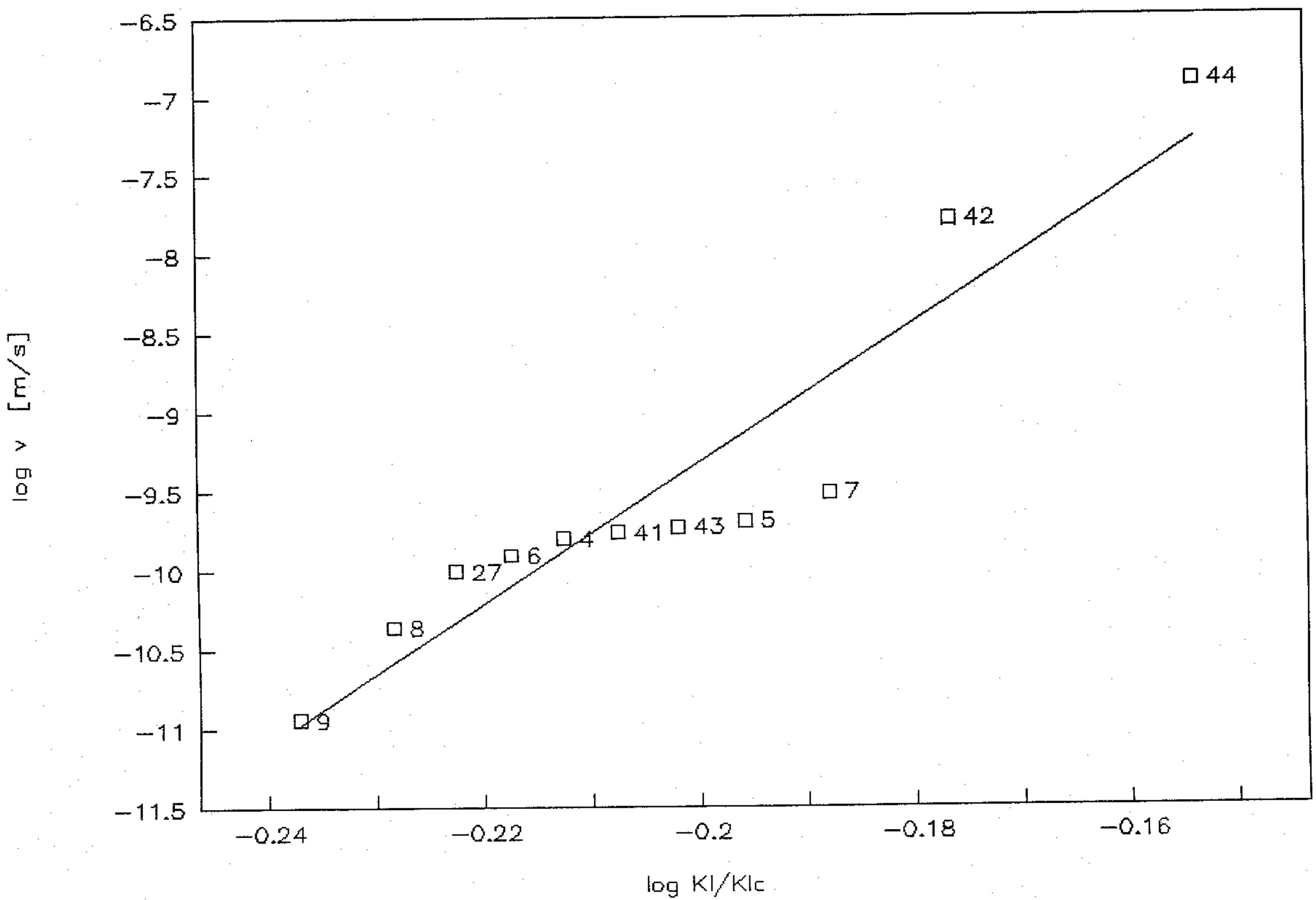

Fig. 3.7. Subcritical crack growth curve for the σ=165 MPa serie.

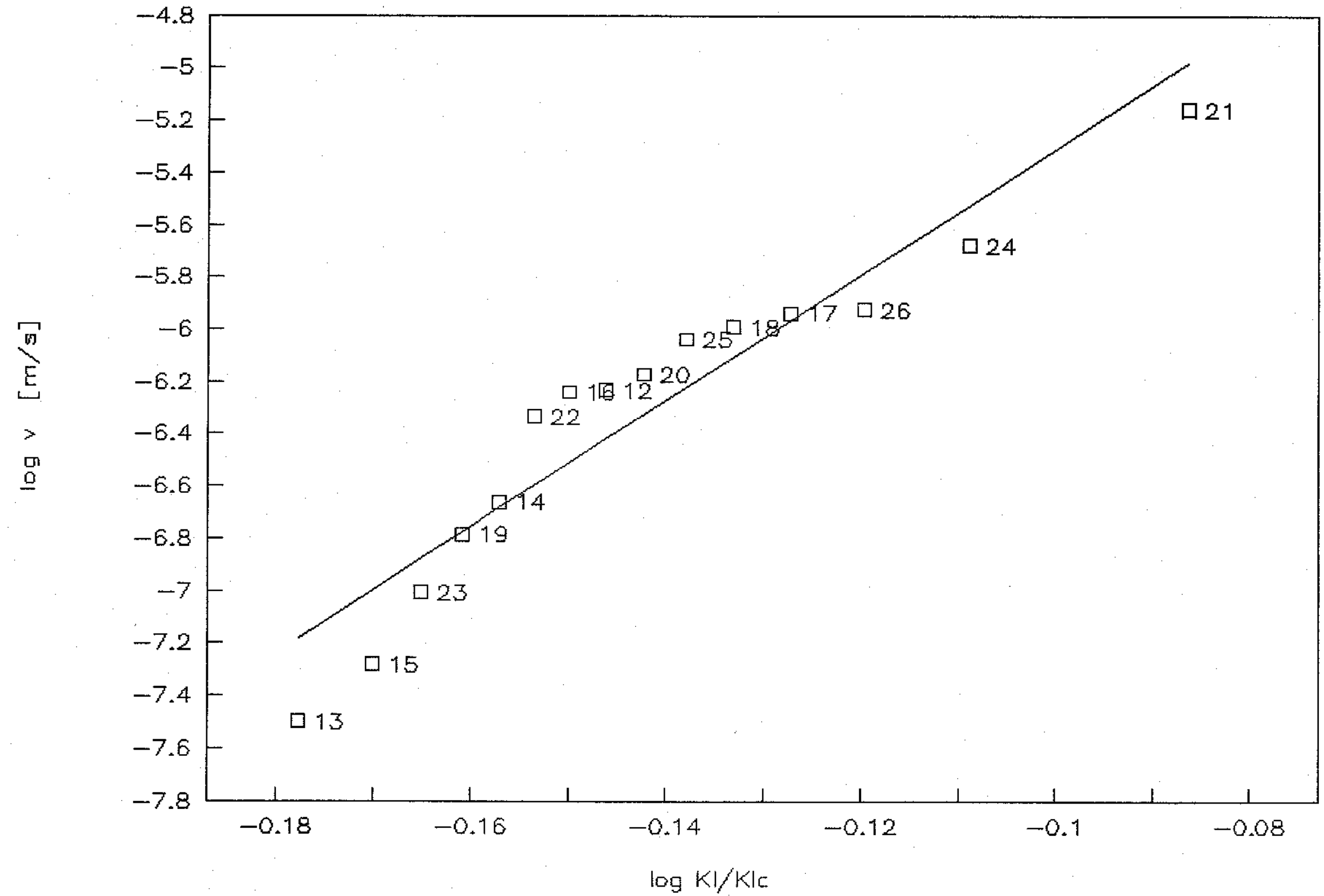

Fig. 3.8. Subcritical crack growth curve for the σ=180 MPa serie.

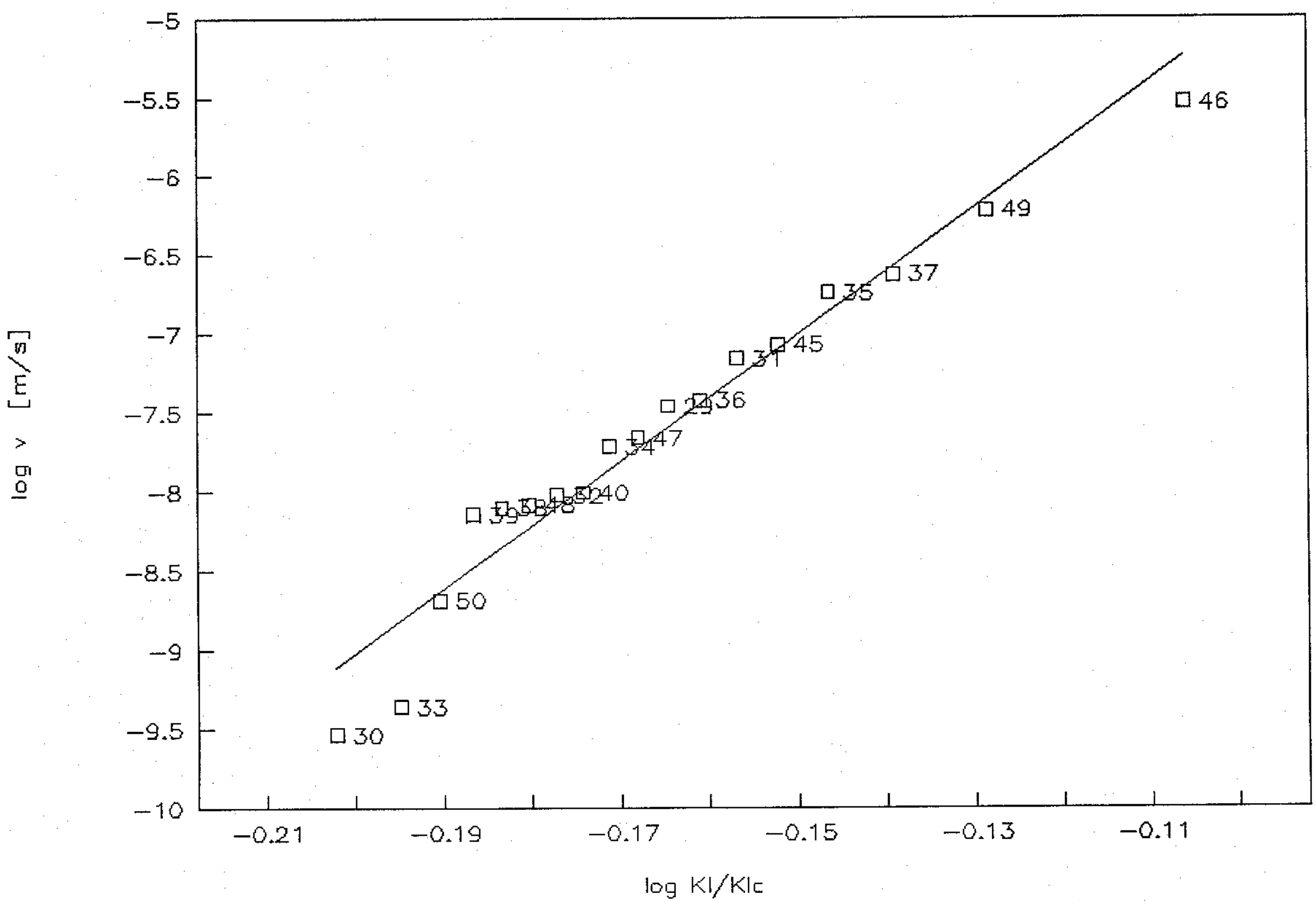

Fig. 3.9. Subcritical crack growth curve for the σ=190 MPa serie.

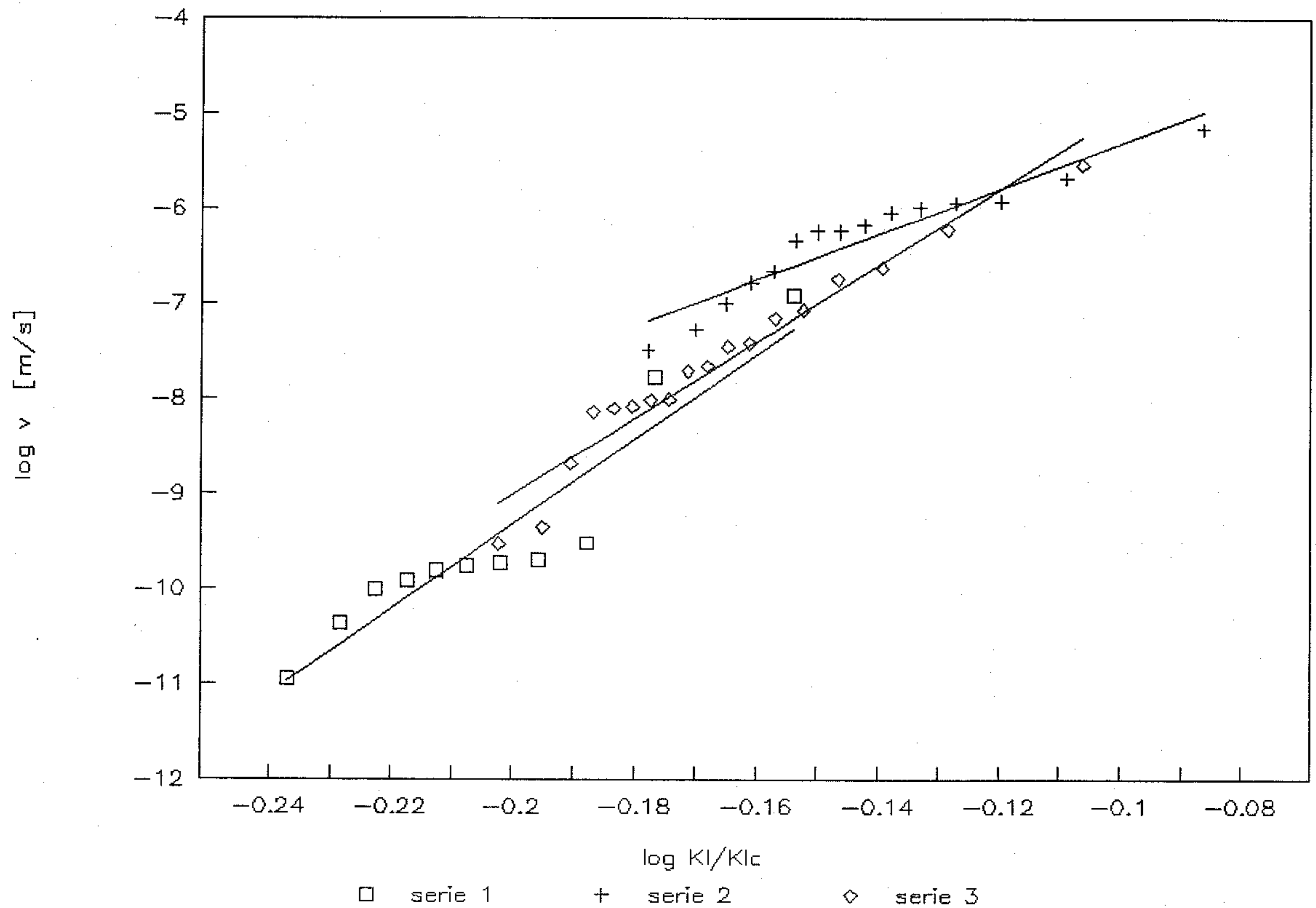

Fig. 3.10. Subcritical crack growth curves of the three series.

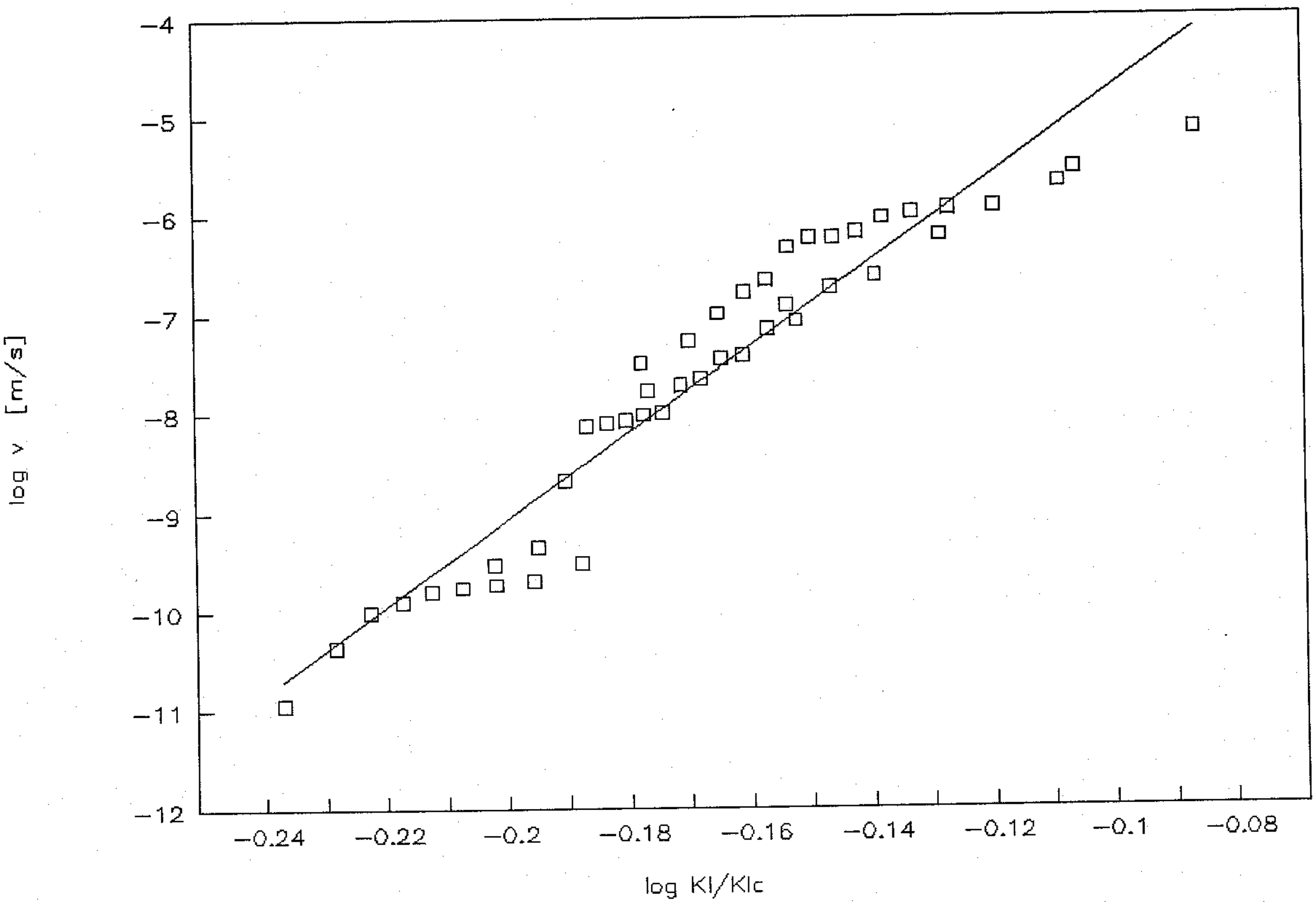

Fig. 3.11 Subcritical crack growth curve of all the data points.