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The Influence of microstructure on the fracture statistics of polycrystalline diamond and polycrystalline cubic boron nitride

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Abstract

Flexural Strength data of a number of grades of polycrystalline diamond (PCD), and polycrystalline cubic boron nitride (PCBN) were analysed using Weibull, normal and lognormal distributions. The role of microstructure in the failure mechanism of such material was analysed in terms of the chosen strength distributions. The best-fit distributions were determined using the maximum log-likelihood criteria and a comparison between the best and worst fit was conducted using the Akaike Information Criteria (AIC). Both large and small specimens were tested to investigate possible volume scaling effects for these materials. The different microstructures between the two materials was shown to have an effect on the statistical strength distributions. It was found that for PCD, in general, a lognormal distribution provided a better fit than the other distributions and no specimen size effect was observed. For PCBN a significant specimen size effect was observed and

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this also corresponded to the data fitting to a Weibull distribution.

Keywords: PCD, PCBN, flexural strength, strength statistics

1. Introduction

Polycrystalline Diamond (PCD) and polycrystalline cubic boron nitride (PCBN) are two grades of superhard materials which are frequently used in the most demanding cutting operations such as rock drilling in the oil and gas industry and the machining of hardened steels and abrasive alloys in the aerospace industry. While such materials have favourable qualities such as high hardness and abrasive resistance they are prone to premature failure due to brittle fracture [1].

In general the strength distribution of brittle materials such as these is caused by small crack like flaws, which are homogeneously distributed throughout the specimen microstructure. The variability of these flaws in terms of size, position and orientation with respect to the loading axis is the primary cause of the large scatter in the maximum strength distribution. There are also a number of flaw types which can lead to failure, such as grain boundaries, pores and impurities within the microstructure. As a result of this, the distribution and magnitude of stress concentrators within the microstructure is extremely varied and this can lead to the strength of such brittle materials being poorly defined.

The Weibull distribution has traditionally been employed to describe the strength distribution of brittle materials [2, 3, 4]. This theory assumes a direct correlation between the density of flaws distributed throughout the specimen and the subsequent strength distribution. The fundamental assumption

of this theory is that of the weakest link hypothesis, where specimen failure is due to the failure of the weakest volume element and that there is no interaction between these critical flaws [2]. However, recent evidence has shown that this distribution may not always be the most accurate description of experimentally measured strength data for brittle materials. Lu et al. [5, 6] compared the strength data for three different ceramics and analysed them using the Weibull and normal distributions. They found that for ZnO, the strength data was best characterized by the normal rather than the Weibull distribution. Similar results have also been reported by Basu et al. [7] and Nohut and Lu [8] for a variety of advanced and dental ceramics. Nohut [9], looked into the influence sample size has on the choice of an accurate strength distribution. It was shown that smaller sample sizes tend to overestimate the Weibull modulus and therefore underestimate the materials failure probability. Studies such as these have shown that the non-critical choice of applying the Weibull distribution for describing strength data for brittle ceramics is no longer sufficient, and further statistical analysis is required in order to accurately describe this data.

While many studies have focused on the strength distributions of advanced ceramics, little research has been conducted in this area for superhard materials. Correct statistical characterization is essential in order to avoid misleading strength measurements and establish reliable information with a view to improving the fracture properties of these materials. This may also lead to an understanding into the role of microstructure and the structure property relationship for superhard materials, which is vital in determining dominant failure mechanisms under working conditions.

In this study, flexural strength data for a number of grades of PCD and PCBN are analysed incorporating Weibull, normal and lognormal distribution functions. The grades vary in terms of grain size and second phase material percentage and three different specimen sizes will also be tested. A comparison between the models' 'goodness-of-fit' to the empirical data is performed using the Akaike Information Criterion (AIC) [12].

2. Materials and method

The flexural strength of each sample was tested in accordance with BS-EN 843:1 [15], and was calculated using Eq. (1).

$$\sigma_f = \frac{3P_{in}s}{2bh^2} \quad (1)$$

where, σ_f is the flexural strength, P_{in} is the maximum breaking load, b is the width, h is the height and s is the sample span. The samples were tested at room temperature and were loaded at a constant crosshead displacement rate of 1 mm/min.

The strength of brittle materials can depend on the area of the stressed volume where a larger volume will lead to an increased chance of failure due to a critical flaw. To investigate possible volume scaling effects in the strength of superhard materials a number of different size specimens were tested - these will be referred to as large, small and micro sized specimens. Table 1, summarizes the specimen dimensions used in this study. Three grades of PCD were provided with constant grain size and varying in second phase percentage while one grade of PCBN was supplied with a similar grain size and second phase percentage. Table 2, shows the percentage of second

phase material within the microstructures and the dimensions of the studied specimens.

Table 1: Specimen dimensions used in study

Size (mm)	Length (l)	Width (b)	height (h)
Large	28.5	6.25	4.76
Small	14	6	3
Micro	6	3	0.5

Table 2: Percentage second phase material and specimen size label

Grade	Second phase (%)	Specimen size
PCD A	8.5	Small
PCD B	7.5	Small
PCD C	7.5	Micro
PCBN A	15	Large
PCBN B	15	Small
PCBN C	15	Micro

3. Theory

In this study the Weibull, normal and lognormal distribution functions have been used in fitting the strength data for the materials tested.

3.1. Weibull Distribution

The Weibull distribution of strength data is based upon the weakest-link hypothesis and the fact that the flaw distribution is homogenous throughout

the specimen microstructure. That is to say that failure of any flaw will lead to complete failure of the material and that there is no interaction between flaws prior to flaw initiation. For the Weibull distribution, the cumulative failure probability of a material which is subjected to a uniform stress (σ) is given by:

$$F(\sigma) = 1 - \exp\left(-\left[\frac{(\sigma - \sigma_{th})}{\sigma_0}\right]^m\right). \quad (2)$$

where, σ_0 is the normalized Weibull strength, σ_{th} is the threshold strength below which no fracture will occur (this is commonly taken to be zero for brittle material where there is a remote possibility of a very large flaw existing within the specimen), and m is the shape parameter for the Weibull distribution. A low Weibull modulus, m indicates a wide scatter in the strength data and for brittle materials this value is in the order of 10 [13]. From this the probability density function can easily be found and is given by:

$$f(\sigma) = \frac{m}{\sigma_0} \left(\frac{\sigma}{\sigma_0}\right)^{m-1} \exp\left(-\left[\frac{\sigma}{\sigma_0}\right]^m\right). \quad (3)$$

where, $f(\sigma) = dF(\sigma)/d\sigma$.

3.2. Normal Distribution

The most widely used specific distribution for describing empirical data is the normal distribution. The normal distribution is described in terms of the mean $\bar{\sigma}$ and the standard deviation α of the materials data and normalized to give its probability density function:

$$f(\sigma) = \frac{1}{\sqrt{2\pi}\alpha} \exp\left[-\frac{(\sigma - \bar{\sigma})^2}{2\alpha^2}\right]. \quad (4)$$

The normal distribution can be used for values close to the mean, so for distributions which exhibit a symmetrical trend this may be a suitable choice. However, it is worth noting that for strength values far below the mean it gives a finite probability of negative strength values occurring [4]. This is an obviously unrealistic outcome which imposes limitations on the use of the normal distribution function.

3.3. Lognormal Distribution

A random variable is defined as being lognormally distributed if the logarithm of that random variable is normal distributed. The probability density function is therefore similar to that of the normally distribution:

$$f(\sigma) = \frac{1}{\sqrt{2\pi}\alpha\sigma} \exp \left[-\frac{(\log(\sigma) - \bar{\sigma})^2}{2\alpha^2} \right] \quad (5)$$

The mean m and variance v of a lognormal random variable are functions of $\bar{\sigma}$ and α that can be calculated by $\exp(\bar{\sigma}^2 - 1) \exp(2\alpha + \sigma^2)$ and $\exp(\alpha + \bar{\sigma}/2)$ respectively [14].

4. Best-Fit Procedures

In order to choose the distribution that most closely represents the empirical data some ‘goodness-of-fit’ test must be performed. The Maximum Likelihood Estimation (MLE) method is a procedure to estimate the parameters of statistical models [10]. The method highlights the smallest coefficient of variation and uses the likelihood of a particular distribution’s density function to determine the model that most represents experimental results [11].

The distribution that yields the largest likelihood is chosen. For a distribution described by its density function $f(\sigma_i)$, the likelihood function is defined as:

$$L = \prod_{i=1}^N f(\sigma_i), \quad (6)$$

and for its loglikelihood is:

$$\ln L = \sum_{i=1}^N \ln f(\sigma_i), \quad (7)$$

where, N is the number of sample repeats. The maximum log-likelihood estimation is the numerical maximization of this function.

This can be further extended to draw comparison between competing models. This is done using the Akaike Information Criterion (AIC) [12]. The AIC is defined by:

$$AIC = -2 \ln \hat{L} + 2k. \quad (8)$$

where $\ln \hat{L}$, is the maximum log-likelihood for a given distribution and k is the number of independently adjusted parameters within the model, for example $k = 2$ when evaluating a two-parameter Weibull distribution. The AIC estimates the mean log-likelihood of a model and provides a versatile procedure for statistical model identification. This simple procedure selects a model with the minimum AIC among several competing models. For a model to show a significantly better fit over other models the difference between them must be greater than 2, that is $\Delta AIC = AIC_{max} - AIC_{min} \geq 2$ [5], where AIC_{min} is the model with the best fit.

5. Results and Discussion

Table 3 presents the parameters for each of the fitted distributions from the experimentally determined strength data. Each material tested contained at least 15 specimen repeats.

Table 3: Fitted parameters for Weibull, normal and lognormal distributions

Material	Weibull		Normal		Lognormal	
	m	σ_0	μ	σ	m	v
PCD A	13.35	1074.70	1033	100.85	1033.50	106.78
PCD B	8.27	1034.30	980.15	121.79	980.45	120.25
PCD C	6.63	1080	1008	173.26	1010.10	176.63
PCBN A	14.28	438	424.84	30.23	424.90	29.96
PCBN B	22.12	624.11	609.21	33.12	609.28	33.20
PCBN C	7.52	789.98	737.89	139.41	740.83	156.38

Table 4: Log-likelihood (MLE) values for Weibull, normal and lognormal distributions and the Akaike Information Criterion for the best fit distribution, $\Delta AIC = AIC_{max} - AIC_{min}$

Material	MLE_w	MLE_n	MLE_{ln}	ΔAIC
PCD A	-395.03	-396.46	-397.45	4.83
PCD B	-401.74	-400.24	-399.76	3.97
PCD C	-244.43	-244.17	-244.13	0.610
PCBN A	-280.57	-279.15	-278.96	3.26
PCBN B	-280.42	-280.52	-280.61	0.366
PCBN C	-160.25	-160.87	-161.53	2.56

PCD grades A and B were of the same specimen dimensions and differed only in percentage second phase material. Comparing the results between these two grades it was observed that a decrease in the second phase material within the microstructure lead to a decrease in the measured characteristic and average strength for each of the fitted distributions. PCD A, which had the highest percentage second phase, also shows the lowest strength scatter among the PCD grades, having the largest Weibull moduli and the lowest additive and multiplicative normal and lognormal standard deviations respectively. Figure 1(a), plots the empirical survival function against the fitted survival function for PCD A. From this it is observed that the Weibull distribution is a better fit for the the data than either the normal or lognormal distributions for this case.

Micro-sized samples of PCD C were tested in order to investigate possible scaling effects when compared to PCD B. From the data presented the strength of both the large and micro samples is quite similar showing little size effect. For Weibull distributed data smaller specimens statistically should fail at higher average loads. This is due to the likelihood of finding a critical destructive flaw in larger specimens being greater than that in smaller ones [16]. The empirical data shows no apparent size effect for the strength of the tested PCD grades, a result that has been noted previously by Lu et al. in the case of zinc oxide [6].

As well as the absence of a size effect, the Weibull moduli for the PCD grades are quite low. Typical modern ceramic materials usually have moduli of between $m = 10 - 20$. While PCD A has a moduli of 13.35, both PCD B & C have low moduli typical of materials with a large scatter in their strength

data (Table 3). A low modulus indicates a low reliability in the strength data as described by the Weibull distribution. Plots of the survival functions against the empirical data for PCBN B and C are shown in Fig. 1(b) and 1(c), both show that the lognormal distribution fits the experimental data better than that of the Weibull distribution.

The Weibull distribution is based on the weakest link hypothesis, that is that the microstructure contains sparsely distributed flaws and it is the largest of these flaws that initiate failure. Weibull analysis assumes that there is no interaction of flaws before failure. For the PCD materials whose microstructure is very complex this may not be the case. The microstructure is composed of a network of interlocking hard grains. These grains can penetrate into one another forming grain/grain bonds or be separated by a trapped pools of second phase material. A typical micrograph for PCD is shown in Fig. 2. When analysing complex microstructures it is often difficult to distinguish where initiation takes place, from a grain boundary or from a pool. Failure from one or another will result in a different strain distribution within the microstructure and therefore fracture occurs at varying loads. It is likely that there are complex interactions between the pools and grains within the microstructure and that initiation is not a result of a single major flaw as is the hypothesis of the Weibull distribution, but rather a combination of a number of interacting factors.

Table 4, shows the maximum log-likelihood (MLE) for each material tested and their corresponding ΔAIC . The best fit distribution is indicated by the smallest negative value for the maximum log-likelihood. Based on the maximum log-likelihood criteria the lognormal distribution is shown to be

statistically the optimal model for both PCD B & C. In the case of PCD A the scatter of strength data was small and therefore it is not surprising that a Weibull distribution describes the data the best. For PCD A and PCD B, the ΔAIC value is greater than 2, which identifies that the dominant model characterizes the strength data significantly better than the competing models, and these results are further illustrated in Fig. 1(a) and 1(b). For PCD C, the ΔAIC value is less than 2 and as stated previously, in order for the AIC criteria to show a significantly better fit for one distribution over another this value should be greater than 2. For values $\Delta AIC < 2$ it is difficult to distinguish which distribution function is most accurate as described by this criteria. However, evidence from Fig. 1(c) suggests that the lognormal distribution fits the empirical data more closely than either the Weibull or normal distributions.

The three PCBN grades in this study are all similar in terms of grain size and second phase material and percentage, differing only in specimen dimensions. Table 3 shows clear size effect with an increase in strength corresponding to a decrease in specimen dimensions. This is further illustrated in Fig. 3, where the mean strength decreases for PCBN with an increase of the effective volume of the tested specimen. The existence of a size effect in strength data for brittle material is well known to be a direct consequence of data set being characterised by a Weibull distribution. Showing that such a size effect exists for PCBN is significant in determining whether the strength data is most accurately described by this distribution. Both PCBN A & B, have a high modulus which further illustrates that strength data of this material may be accurately described by the Weibull distribution. PCBN C

has a modulus of less than 10, which indicates a higher degree of scatter in strength data for the micro-sized samples. It has been previously noted by Danzer et al. [18] that for small samples, when failure is initiated from very small flaws whose density is high within the microstructure strong interactions between these flaws exist, and the Weibull theory may fail to accurately fit the data.

Table 4, details the MLE and AIC criteria results, and for two of the three grades (PCBN B & PCBN C) suggests that a Weibull distribution is the most successful fit to the empirical data. It should be noted however that the ΔAIC for PCBN B is quite low. The empirical survival functions are given for the PCBN grades in Fig. 4. For PCBN B, the Weibull distribution is plotted as the solid line and is clearly a better fit to the empirical data than the other competing models. This evidence, along with the high modulus for PCBN B indicates that indeed the Weibull function is the most appropriate fit for the PCBN B grade.

Fig. 5, presents a typical PCBN microstructure used in this study. The dark phase is the CBN grains which are distributed throughout the microstructure. The microstructure of PCBN is very different to that of PCD and its effect on the statistical distribution of the strength data has been shown above where, for PCBN strength has been predominantly best fit by a Weibull distribution as opposed to a lognormal fit for the PCD specimen. Unlike PCD, the grains in PCBN are not interconnected but surrounded by the second phase matrix. For this type of microstructure it is difficult for interactions between grains and the second phase matrix to occur before initiation. This contrasts with PCD, whose microstructure of interlocking grains

increases the likelihood of micro-cracks forming. Prior to crack initiation it is likely that these flaw will undergo some crack bridging and interaction between critical flaws will be high. For PCBN flaw density is relatively low, as therefore little homogenization of critical flaws will occur but rather failure from one major flaw within the microstructure. This is especially true for low rate testing where it has been previously shown that fracture is predominantly inter-granular [17]. This type of specimen failure is the main hypothesis of the Weibull distribution and it is therefore no surprise that it best describes the strength distribution of PCBN.

6. Conclusion

Strength data for two superhard materials were investigated using three statistical distributions. Based on limited sample sizes a minimum information criterion was used to determine the best fit distribution for each material. It was observed that for PCD the Weibull distribution was not the most appropriate model to characterize the strength data which was more closely described by a lognormal distribution. For smaller sample dimensions, little deviation in the maximum failure strength was found, a result that further supports the distribution not being Weibull in nature. For PCBN a significant size effect was observed and using the minimum information criterion a Weibull distribution was found to be the best fit. The role of microstructure of superhard materials has been shown to cause a change in the statistical distribution of measured strength data. Applying correct statistical distributions is vital in determining accurate failure probabilities for the materials under investigation. Understanding the distribution associated with strength

data for a particular material also provides a useful insight into the material's dominant failure mechanism and provides a framework for determining accurate models of the structure-property relationship that exists for superhard materials.

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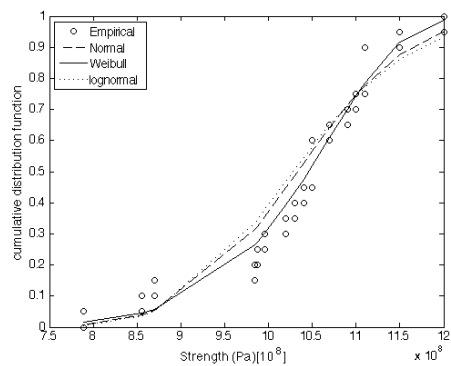
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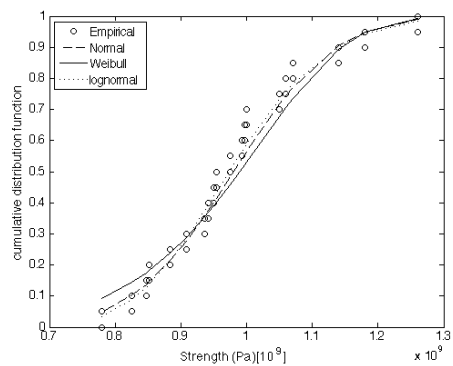
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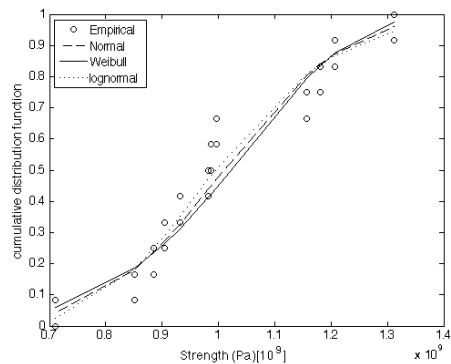
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(a)



(b)



(c)

Figure 1: Empirical and fitted survival functions for (a) PCD A, (b) PCD B, and (c) PCD C

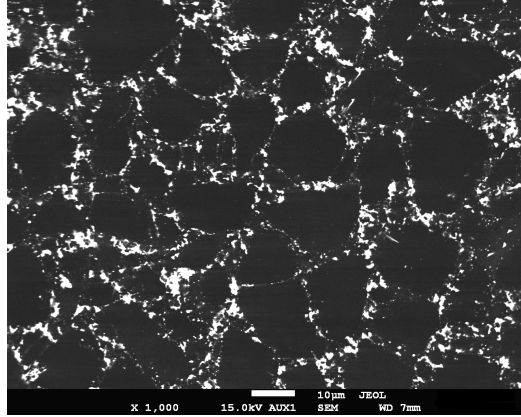


Figure 2: Scanning electron micrograph of typical PCD sample used in test

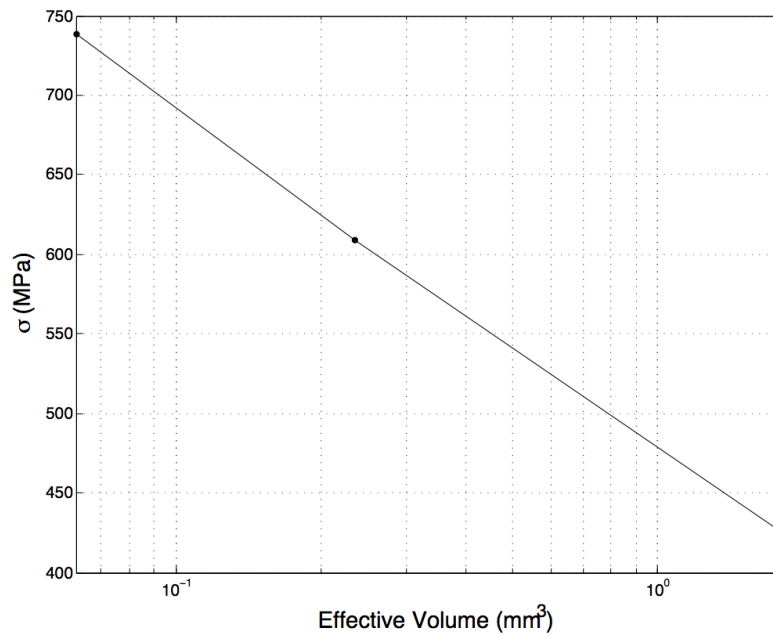
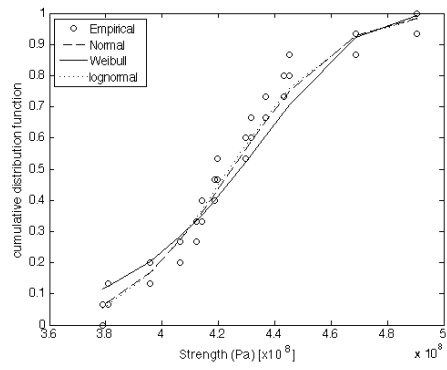
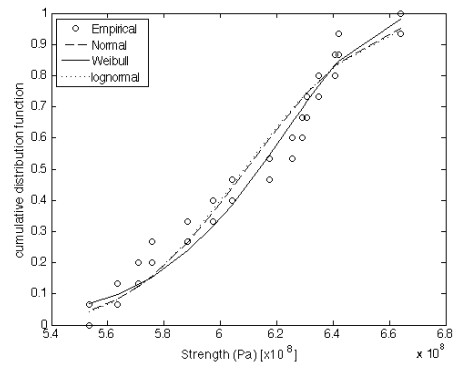


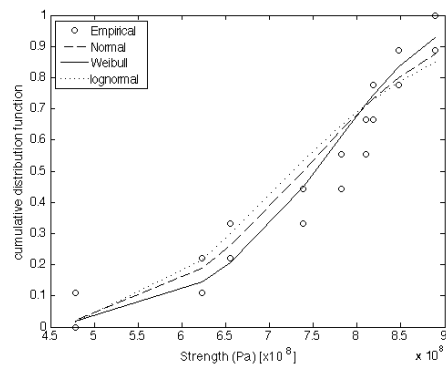
Figure 3: Volume size effect for PCBN on the mean strength



(a)



(b)



(c)

Figure 4: Empirical and fitted survival functions for (a) PCBN A, (b) PCBN B, and (c) PCBN C

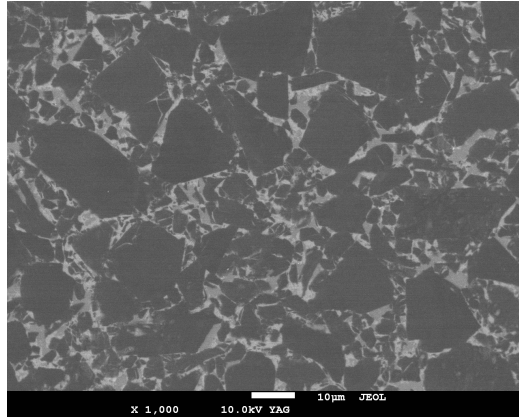


Figure 5: Scanning electron micrograph of typical PCBN sample used in test

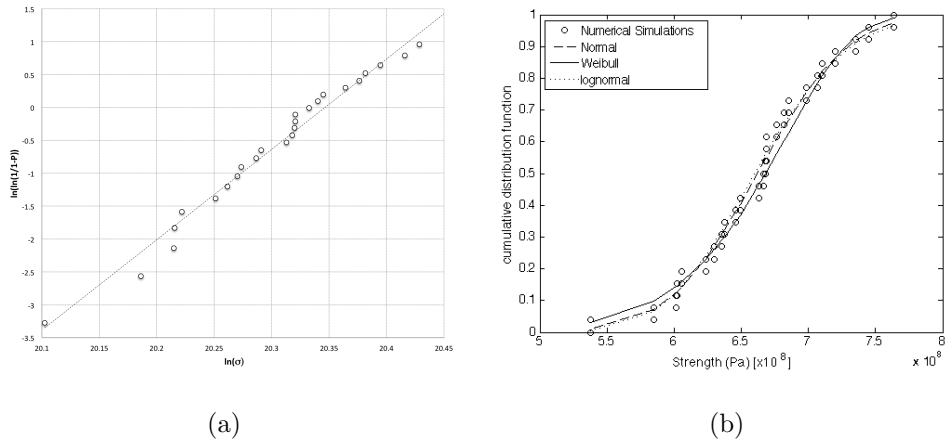


Figure 6: Strength distribution for simulations (a) fitted on a Weibull plot and, (b) against the cumulative density function