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# FRACTURE STATISTICS USING THREE-PARAMETER AND TWO-PARAMETER WEIBULL DISTRIBUTIONS FOR Fe-0.4C-1.5Cr-1.5Ni-0.8Mn-0.2Mo STRUCTURAL SINTERED STEEL

A statistical analysis is presented of tensile and bending strengths of a porous sintered structural steel which exhibits non-linear, quasi-brittle, behaviour. It is the result of existing natural flaws (pores and oxide inclusions) and of the formation of fresh flaws when stress is applied. The analysis is by two- and three-parameter Weibull statistics. Weibull modulus, a measure of reliability, was estimated by the maximum likelihood method for specimen populations < 30. Probability distributions were compared on the basis of goodness to fit using the Anderson-Darling tests. The use of the two-parameter Weibull distribution for strength data of quasi-brittle sintered steels is questioned, because there is sufficient evidence that the 3-parameter distribution fits the data better.

Keywords: sintered structural steels, mechanical properties, Weibull statistics, maximum likelihood, threshold parameter

# 1. Introduction

Ductile wrought steels are widely used in engineering structures. They usually can sustain a lot of plastic damage before failing. The stress concentrations which may arise in a material are redistributed through local plastic flow. We understand quite well how tolerant wrought steels are to damage and the ways that such alloys fail, so efficient structures may be designed with well-defined margins of safety or reserve strength. By comparison, a brittle material behaves elastically up to the maximum load at which catastrophic failure occurs and can fail without prior warning so much larger safety margins are needed. White cast iron and sintered carbides are the most common example of such fracture. Brittle fracture is controlled by microscopic inclusions, surface and interior flaws and defects, as well as pores of different sizes and shapes present in the sintered material arising from the manufacturing process.

An intermediate category of fracture, however, generally known as quasi-brittle fracture, needs to be defined. A quasi-brittle engineering material, a title which encompasses many sintered and hardened, sinterhardened and sinteraustempered steels, shows measurable deformation prior to failure. Material-related factors influence the mechanical behaviour more in quasi brittle than in ductile materials and consequently strength tests on many quasibrittle sintered steels usually show wider variations. For sintered structural steels tested in tensile and bend tests experimental studies consistently reveal large scatter in the measured values of fracture strength [1-3]. The mechanical properties of these steels are strongly influenced by material composition, processing method and surface state. Specimens made of a sintered steel have variable crosssectional areas (the effect of porosity), and variable strength of the metal matrix. A sintered steel specimen contains a number of flaws of different sizes and shapes arising from the manufacturing process. Under sufficiently high stress, existing flaws in the quasi-brittle steel start growing and new flaws form; in some steels at the beginning of nonlinearity in the load-displacement relationship, in others earlier or later [4]. Furthermore for porous sintered steels the stressstrain diagram may be nonlinear from initial loading until final rupture. During plastic deformation stable microcrack coalescence and microcrack growth in the material before the final fracture has been reported [5]. Quasi-brittle PM steels fail before the point of plastic instability is reached, i.e. stress for cracking is smaller than for further plastic deformation. The peak load corresponds to failure stress by cracking, not the stress at the point of plastic instability: (U)TS.

The microstructural factors in PM steels include powder particle size, different pore sizes, shapes and distribution, or the irregular distribution of the constituents of the heterogeneous sintered material. These factors ensure that the strength of the sintered specimen/element is not a feature that is treated deterministically, but should be treated in a probabilistic manner and expressed by a function. For this reason, porous sintered steels which fracture in a quasi-brittle manner should be evaluated statistically [6, 7]. Peirce [8] was the first to formulate a weakest link theory, in which the fracture of a specimen is identified with the unstable propagation of the most "critical" flaw (the largest in a uniform stress field) and recognized the close relation of this model to the theory of extremes values, stating that the distribution of smallest values tends in the limit of large number of specimens to be one of the asymptotic distributions, regardless of the initial population [9].

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The mathematical basis for probabilistic treatment was developed by A. M. Freudenthal and E. J. Gumbel [10] according to suggestions of W. Weibull [11]. However, at present, the applicability of the Weibull distribution to describe the fracture strength of quasi-brittle materials has been a subject of some debate.

Predictive procedures for flaw assessment require a robust and verified methodology which reflects the strong role of microstructural constraints and statistical variability on correlations of strength data for varying flaws configurations and loading modes (tension vs. bending) [12].

#### 2. Experimental procedures

Höganäs Astaloy CrL pre-alloyed powder, Elkem lowcarbon ferromanganese (77%Mn, 1.3%C, < 40µm), nickel (>40µm) and ultra fine Höganäs CU-F graphite powders were the starting materials in this investigation. Nickel was admixed at 1.5%, ferromanganese at 0.8% Mn and graphite at 0.4%. The composition of the test material is shown in Table 1.

TABLE 1 Chemical composition of tested steel (wt. %)

| Mixture | Fe   | С   | Cr  | Ni  | Mn  | Mo  |
|---------|------|-----|-----|-----|-----|-----|
| 34 HNM  | 95.6 | 0.4 | 1.5 | 1.5 | 0.8 | 0.2 |

Mixes were made in a Turbula mixer, for 60 minutes. No lubricant was added. The powders were single-action uniaxially compacted into ISO 2740 dog-bone tensile test bars in a steel die at 660 MPa using die wall lubrication. This was followed by sintering in a horizontal laboratory furnace, in nitrogen furnace atmosphere, in a semi-closed stainless steel container [13, 14] containing also lumps of ferromanganese (the proportion the weight of ferromanganese lumps to the weight of sintered samples was 5.7%). Compacts were heated to the sintering temperature at a rate of 65K/min, and held at 1120 or 1250°C for 60 minutes. Then, after the sintering process, the compacts were subjected to various variants of cooling. For each variant 25 specimens were prepared. The sintering and heat treating conditions employed in the present study are summarized in Table 2.

Observations of the specimens revealed a microstructure consisting of bainite (SAT 500), bainite and martensite (S+H), pearlite and ferrite (S+C), and nickel-rich regions. The austenite was in the vicinity of the nickel-rich diffusion regions of the iron particles. For the 1250°C sintered specimens, degree homogenization and pore rounding of the microstructure was more pronounced. SEM analysis of the TS and TRS fracture surfaces revealed mixed mode, both transgranular and ductile rupture fracture with a greater amount of transgranular fracture in the case of the 1250°C sintered specimens having the highest level.

Standard PN-EN ISO 2740 specimens were tested in tension and in three-point bending, in both cases undergoing plastic deformation before fracture. Tensile specimens were tested for the tensile strength (TS) on an MTS 810 servo-hydraulic machine at a rate of 1 mm/min. The specimens exhibited non-linear behaviour in tension. Fig. 1 shows stress-strain curve for a SAT 500 1120°C specimen, exhibiting one of the highest stress level achieved in the samples tested.

The same type of specimen was tested in three-point bending to determine the apparent transverse rupture strength, TRS, using roller-type contact points jig with span length, l, of 28.6 mm, at a crosshead speed of 2 mm/min. In all test cases, between 24 or 25 test specimens were evaluated for each thermal cycle. The statistically elaborated properties of the PM steels are summarized in Table 3 as a function of processing history. Some of the test pieces failed within the locating threads rather than within the gauge length. When compared at a fixed thermal cycle, the higher sintering temperature produced higher TS and TRS.

# TABLE 2

| Sintering and | l heat treati | ng condition | and designati | on of the specimens |
|---------------|---------------|--------------|---------------|---------------------|
|---------------|---------------|--------------|---------------|---------------------|

| Process            | Sintering<br>temperature | Cooling after sintering | Isothermal holding in a furnace | Tempering     | Designation |
|--------------------|--------------------------|-------------------------|---------------------------------|---------------|-------------|
| Sinteraustempering |                          | 40K/min.                | 500°C/60 min.                   | -             | SAT 500     |
| Sinterhardening    | 1120 and 1250°C          | 65K/min.                | -                               | 200°C/60 min. | S+H         |
| Sintering          |                          | 10K/min.                | -                               | -             | S+C         |

TABLE 3

The statistically elaborated results of mechanical testing of 34 HNM sintered steel – mean values and standard deviation were measured on 24 or 25 specimens

| Sintering<br>temperature | Specimens<br>designation | Number of specimens, N | TS, MPa      | TRS, MPa       | TRS/TS |
|--------------------------|--------------------------|------------------------|--------------|----------------|--------|
| 1120°C                   | SAT 500                  | 25                     | $644\pm45$   | $1137\pm115$   | 1.76   |
|                          | S+H                      | 24                     | $622 \pm 25$ | $1125\pm76$    | 1.81   |
|                          | S+C                      | 24                     | $508\pm36$   | $947 \pm 123$  | 1.86   |
| 1250°C                   | SAT 500                  | 25                     | $728\pm50$   | $1290\pm128$   | 1.71   |
|                          | S+H                      | 25                     | $710 \pm 35$ | $1319\pm106$   | 1.86   |
|                          | S+C                      | 24                     | $669 \pm 43$ | $1242 \pm 120$ | 1.86   |

# 3. Statistical data reduction

In describing the strength of a brittle material the probabilistic statistical approach invokes that the specimen will fail immediately when the stress intensity at one of the flaws reaches the critical value for cracking – the weakest link concept. Numerous tests confirm the universal character of the Weibull approach [7]. Currently the Weibull distribution is commonly found in materials science, and was confirmed it generally applies even if interaction between flaws is considered.

A continuous probability function derived from weakest link statistics conveniently characterizes the distribution of strengths values of specimens in the form [15]:

$$P_{f} = 1 - P_{s} = 1 - \exp\left[-\left(\frac{\sigma - \sigma_{u}}{\sigma_{r}}\right)^{m}\right] \text{ for } \sigma \ge \sigma_{u} \qquad (1)$$
  
and  $P_{f} = 0$  for  $\sigma < \sigma_{u}$ ,

$$P_{f} = 1 - P_{s} = 1 - \exp\left[-\left(\frac{\sigma - \sigma_{u}}{\sigma_{0} - \sigma_{u}}\right)^{m}\right] \text{for } \sigma \ge \sigma_{u} \qquad (2)$$
  
and  $P_{f} = 0$  for  $\sigma < \sigma_{u}$ ,

where:

$$\sigma_0 - \sigma_u = \sigma_r = \frac{E - \sigma_u}{\Gamma(1 - 1/m)}$$
(3)

- $\Gamma$  is the Euler gamma function and *E* is the expected value of the distribution function;
- m Weibull modulus, the shape parameter;
- $\sigma_0$  characteristic stress at which a fraction 1/e of
- specimens survives the scale parameter;  $\sigma_u$  – threshold stress below which the failure probability is zero – the shift parameter;
- $\sigma_r$  location parameter;
- $P_f$  the failure probability of specimens made of the tested materials subjected to stress  $\sigma$ ,
- $P_s$  the survival probability of test specimens subjected to stress  $\sigma$ .

Three parameter (3-p) Weibull distribution not only models the actual distribution of strength, but also allows us to predict and assess the strength of the weakest specimen of the entire population. The threshold value can be used to determine the minimum value of properties of the designed steel. It also helps to use the obtained results of laboratory specimens for the design of structural elements, where the stress distribution is much more complicated.

Often, the threshold fracture strength is set equal to zero  $(\sigma_u = 0)$ , so that the Weibull function given by Equation (1) assumes its more familiar two-parameter form:

$$P_{f} = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_{0}}\right)^{m_{2}}\right] \text{for } \sigma > 0 \qquad (4)$$
  
and  $P_{f} = 0 \text{ for } \sigma \le 0$ 

Rearrangement of Eq. (4) gives:

$$\ln \left[ -\ln \left( 1 - P_{f} \right) \right] = \ln \ln \left( \frac{1}{P_{s}} \right) =$$

$$m \ln \sigma - m \ln \sigma_{0}$$
(5)

and in practice, rather than showing  $P_f$  versus  $\sigma$  it is advisable to plot  $\ln[-\ln(1-P_f)]$  or  $\ln\ln(1/P_s)$  versus  $\ln\sigma$ , since this yields a linear dependence with slope *m*.

Therefore, a two parameter (2-p) distribution is a particular case of a 3-p Weibull distribution which implicitly defines a zero threshold stress for fracture. Consequently, stresses vanishingly small compared to the fracture stress yield a non-zero (albeit small) probability for fracture.

A key feature of this approach is that local fracture stress  $\sigma$  follows a distribution characterized by the Weibull modulus, *m*, and the scale parameter,  $\sigma_0$ , both depending on the interaction between the distribution of flaws and the stress field. Here, the Weibull modulus, *m*, plays a major role in the process to correlate effects of constraint loss for varying flaws configurations and loading modes. In the case of quasi-brittle sintered metal, the parameter  $\sigma_u$  usually take values greater than zero. To eliminate this apparent drawback with a zero threshold stress, a number of researchers advocate the use of 3-p Weibull distribution.

There is a number of methods for determination of Weibull parameters from mechanical strength measurements. Zanakis in his paper [16] documents 17 different methods of obtaining the parameters of the 3-p Weibull distribution, but only two are in common use: the method of maximization of probability and the popular method of linear regression (least squares method) [17-20]. Very good fit of  $\sigma_u$ ,  $\sigma_0$  and m parameters can be obtained using the MLE (Maximum Likelihood Estimation Method) method, which indicates the smallest coefficient of variation (ratio of standard deviation and mean value of a random variable) [3]. This method allows us to find the  $\sigma_u$ ,  $\sigma_0$  and m parameters, and predict with the largest probability, the distribution of fracture strength values of tested specimens. The MLE method is a popular statistical method, which is also the best way to fit a mathematical model to experimental data. Data modeling using the MLE method allows us to adjust the three model parameters to ensure the best fit. The advantage of this method is that it provides a minimum estimation error in the case of analyzing the lowest and highest strength values. In the case of an insufficient number of tested specimens may, however, lead to serious errors in estimating the value of the m parameter. Abnormally low or high strength values may be the result of the improper alignment of specimen in jaws of a testing machine or local friction in roller-type contact points jig in the bend test, which is a major drawback.

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# 3.1. Estimation of the Weibull parameters

Although the parameters for the 2-p Weibull distribution could be determined using a least-squares fit with a weight function on the linearized Weibull equation (linear regression – LR), the MLE method was used in this investigation. For known experimental failure data  $\sigma_i$  (i = 1, 2, ..., N). the parameters  $\sigma_u$ ,  $\sigma_r$  and m were determined by maximization of the likelihood probability density function:

$$L = \prod_{i=1}^{N} f(\sigma_i; \sigma_u; \sigma_r; \mathbf{m})$$
(6)

For calculation of the three parameters,  $\sigma_r$ ,  $\sigma_u$  and m from the 3-p Weibull distribution, the following equations were used:

$$N\sigma_0^m - \sum_{i=1}^N (\sigma_i - \sigma_u)^m = 0$$
<sup>(7)</sup>

$$\frac{N}{m} - \sum_{i=1}^{N} \left[ \left( \frac{\sigma_i - \sigma_u}{\sigma_r} \right)^m \ln \left( \frac{\sigma_i - \sigma_u}{\sigma_r} \right) \right] - \sum_{i=1}^{N} \ln \left( \frac{\sigma_i - \sigma_u}{\sigma_r} \right) = 0$$
(8)

$$\left(1-\frac{1}{m}\right)\sigma_r^m\sum_{i=1}^N\frac{1}{\sigma_i-\sigma_u}-\sum_{i=1}^N(\sigma_i-\sigma_u)^{m-1}=0$$
(9)

While eqns. (7, 8, 9) are non-linear, they have a unique positive

solution, and were solved by the Newton–Rhapson iteration technique. MLE method gave a biased estimate of the Weibull modulus.

The estimators of the 2-p Weibull parameters, *m* and  $\sigma_0$ ; should satisfy the following equations [21, 22]:

$$\frac{\sum_{i=1}^{N} \sigma_{i}^{m} \ln \sigma_{i}}{\sum_{i=1}^{N} \sigma_{i}^{m}} = \frac{1}{m} + \frac{1}{N} \sum_{i=1}^{N} \ln \sigma_{i}$$
(10)

where *m* can be obtained by an iterative procedure and then  $\Sigma_0$  is calculated by

$$\sigma_0^m = \frac{1}{N} \sum_{i=1}^N \sigma_i^m \tag{11}$$

Supposing that there is a random experimental scatter, it can be shown that there are two such cases out of three, which genuine m value is in the range

$$\pm \frac{m}{\sqrt{2N}} = \text{standard error}$$
(12)

where, N is the number of specimens.

Equations (1) and (2) can be used in two ways: to estimate the parameters of the Weibull distribution based on experimental data or, when the parameters are already known - for the analysis of the likelihood of the failure of

TABLE 4

Descriptions of data sets parameters for 2-p and 3-p Weibull distributions, p-values and LRT

|                     |                | 2-p Weibull    |                 |       | 3-p Weibull |      |                         |                         |                 |                   |       |
|---------------------|----------------|----------------|-----------------|-------|-------------|------|-------------------------|-------------------------|-----------------|-------------------|-------|
| Processing variant  | т              | $\overline{m}$ | $\sigma_0,$ MPa | A-D   | P-v (A-D)   | т    | σ <sub>r</sub> ,<br>MPa | σ <sub>u</sub> ,<br>MPa | A-D<br>eq. (13) | p-values<br>(A-D) | LRT   |
| SAT 500 1120 TS     | 17.56<br>±2.48 | - 13.94        | 663             | 0.394 | >0.250      | 8.99 | 346                     | 316                     | 0.352           | 0.374             | 0.735 |
| SAT 500<br>1120 TRS | 10.32<br>±1.46 |                | 1190            | 0.736 | 0.048       | 2.04 | 248                     | 917                     | 0.398           | 0.391             | 0.021 |
| S+H<br>1120 TS      | 26.04<br>±3.76 | 21.21          | 634             | 0.662 | 0.078       | 2.81 | 71                      | 600                     | 0.298           | >0.500            | 0.042 |
| S+H<br>1120 TRS     | 16.58<br>±2.37 | - 21.31        | 1159            | 0.309 | >0.250      | 3.53 | 260                     | 891                     | 0.167           | >0.500            | 0.174 |
| S+C<br>1120 TS      | 17.41<br>±2.51 | - 12.83        | 524             | 0.249 | >0.250      | 8.30 | 256                     | 266                     | 0.266           | >0.500            | 0.785 |
| S+C<br>1250 TRS     | 8.25<br>±1.19  |                | 1000            | 0.351 | >0.250      | 2.73 | 341                     | 643                     | 0.212           | >0.500            | 0.086 |
| SAT 500 1250 TS     | 16.15<br>±2.28 | - 13.97        | 750             | 0.366 | >0.250      | 3.97 | 189                     | 556                     | 0.290           | >0.500            | 0.182 |
| SAT 500<br>1250 TRS | 11.80<br>±1.67 |                | 1345            | 0.300 | >0.250      | 5.53 | 642                     | 698                     | 0.336           | 0.425             | 0.488 |
| S+H<br>1250 TS      | 23.01<br>±3.25 | - 18.19        | 726             | 0.535 | 0.174       | 5.42 | 176                     | 548                     | 0.365           | 0.358             | 0.300 |
| S+H<br>1250 TRS     | 13.37<br>±1.89 |                | 1367            | 0.381 | >0.250      | 2.63 | 283                     | 1067                    | 0.143           | >0.500            | 0.064 |
| S+C<br>1250 TS      | 19.50<br>±2.81 | 15 20          | 688             | 0.841 | 0.025       | 9.54 | 346                     | 341                     | 0.857           | 0.010             | 0.895 |
| S+C<br>1250 TRS     | 11.09<br>±1.60 | 15.29          | 1296            | 0.361 | >0.250      | 2.51 | 309                     | 968                     | 0.123           | >0.500            | 0.057 |

other components. In the latter case, the analysis is usually preceded by the finite element method, determining the stress distribution in the material. The mentioned examination methods (maximum likelihood) for the calculation of 3-p Weibull distribution estimators allow us to calculate  $\sigma$ u parameter, specifying - in some way - threshold strength of the quasi-brittle material. Descriptions of data sets parameters for 2-p and 3-p Weibull are given in Table 4. Fig. 2 shows 2-p and 3-p Weibull plots for data sets of tensile strength of SAT 500 1120 TS specimens.

## 3.2. Goodness of fit

In this study the calculations with the Anderson-Darling (A-D) test for testing the 2-p and 3-p Weibull distribution was used. The A-D test is defined as:

$$A^{2} = -\left\{ N + \frac{1}{N} \sum_{i=1}^{N} (2i-1) \left[ \ln F_{0}(\sigma_{(i)}) + (2N+1-2i) \ln \left(1-F_{0}(\sigma_{(i)})\right) \right] \right\}$$
(13)

Where  $F_{\theta}$  is the assumed (Weibull) distribution with the assumed or specimen estimated parameters.  $\sigma_{(i)}$  is the i-th sorted, standardized, specimen value ( $\sigma_{(i)}$  – order statistics), N – is the specimen size.

Another quantitative measure for reporting the result the A-D test is the p-value. We can calculate p-value for known  $A^2$ . We use the corresponding p-value to test if the data come from the Weibull distribution (Table 4). If the p-value is less than a chosen level of significance (usually 0.05), the null hypothesis that the data come from that distribution should be rejected.

Likelihood ratio tests (LRT) have also been used to compare two distributions (Table 4). LRT compares the fit of 2-p Weibull distribution with the fit of 3-p distribution. If 3-p Weibull distribution significantly improves the fit, the p-value for LRT statistics will be very small [23].



Fig. 1. Stress-strain curve for a SAT 500 1120°C specimen, exhibiting one of the highest stress level achieved in the samples tested



Fig. 2. 2-p and 3-p Weibull plots for data sets of tensile strength; calculated Anderson-Darling and P-values; 95 % confidence intervals

#### 4. Discussion

Data presented in Tables 3 show that the results of a strength tests strongly depend on the specimens' thermal cycle. Analyzing the results of the investigations, it can be seen that the highest values of TS of steel are obtained after sinteraustempering, independently of the sintering temperature. After sintering in 1250°C, sinterhardening and tempering (S+H variant), the TRS value was 29 MPa higher than after sinteraustempering in the same sintering temperature. The lowest mechanical strength values of the investigated steel were observed after sintering + cooling with average rate of 10 K/min thermal cycle. Increasing the sintering temperature from 1120 to 1250°C significantly improves the mechanical properties of Fe- 0.4%C-1.5%Cr-1.5Ni%-0.8%Mn-0.2%Mo-PM steel for all processing variants.

# 4.1. Applicability of Weibull parameters to the quasi-brittle PM steel fracture predictions

The investigated sintered steel is multi-phase, porous, discontinuous and inhomogeneous material, therefore, its mechanical properties frequently exhibit an unusually wide degree of variability. Metallography did not reveal any single clear reason for this. The scatter of PM steel strength has been attributed to a natural statistical distribution of many flaws (pores, oxides, cracks) introduced during manufacturing and loading. On the basis of flaw distribution that is a material characteristic, the reliability of PM steel specimen/structural part strength will exhibit a characteristic statistical variability. This work presents useful formulae to analyse the variability of the mechanical properties of sintered steels.

Several researchers have used the Weibull theory to model quasi-brittle materials. These materials exhibit significant precursory fracture events (crackling noise [24] and non-linear behaviour in tension/bend test) before failure. Numerical calculation reported in [25] show that crack bridging is an important form of crack interaction in the quasi-brittle materials that can significantly alter the resulting Weibull modulus away from the dilute limit (diluted brittle cracks). Therefore, the weakest-link assumption is not accurate for quasi-brittle materials. We show that even if the microscopic strength distribution is Weibull, the emergent distribution is significantly distorted due to elastic interactions, local plastic deformation and metastability.

The Weibull distribution function has been widely used as a statistical tool for predicting the fracture probability in structural materials. However, proper use Weibull distribution requires that its parameters be accurately estimated. Therefore, the 2-p and 3-p Weibull functions were estimated and compared in this study. The Weibull distribution is widely used to describe the scatter of the strength in brittle (but also quasi-brittle) materials, often assuming that the Weibull modulus is a material constant. One possible motivation of this perhaps comes from the classical Freudenthal's interpretation of Weibull modulus depending on the pore size distribution, which however assumes the pores to be at large distance one from the other. However, it was also shown that Weibull modulus, threshold parameter, as well as a geometry and loading condition dependent scale parameter do not necessarily correspond only to alloy properties. Indeed, the Weibull modulus takes into account the stress gradient, but also reflects the influence of the variability in flaw sizes, and can vary because of interaction between the pores or between the cracks and the stress field [26].

Therefore, we postulate that the Weibull modulus, *m*, is a specimen/part or eventually material property but not a material constant. It is a measure of the variability of the fracture strength,  $\sigma_f (\sigma_{TS}, \sigma_{TRS})$  or quantity  $\sigma_f - \sigma_u$ , where  $\sigma_u$  is the threshold stress. Thus high values of m describe a more consistent material. Although  $\sigma_u$  is also clearly a specimen/material dependent parameter, it is often set to zero. This is the safest approach when we have insufficient experimental data to determine su accurately. In effect  $\sigma_u = 0$  states that there is no upper limit to the size of the largest flaw in the material.  $\sigma_0$  is also a material parameter. Essentially, the mean fracture stress increases with  $\sigma_0$ , for given values of *m* and  $\sigma_u$ , as well as specimen shape and volume.

# 4.2. Explaining the discrepancy between Weibull modulus calculated for tensile and bend strength

The most important feature of the results is that they show considerable differences between Weibull moduli for tensile and bend strength. The TRS dispersion evidenced is somewhat higher for all studied materials; and accordingly, the corresponding Weibull analysis yields lower values, indicative of low reliability from a structural viewpoint. We propose an explanation for the different Weibull modulus in bending and tension which is consistent with the progressive, noncatastrophic failure process often observed in bending of semi-brittle PM steel single-action uniaxial compacted specimens with an unsymmetrical density gradient. The microstructure and properties of single-action uniaxial compaction are known to vary with orientation and location. Consequently, the Weibull modulus data obtained from three point bending are lower than that obtained from the tensile test. This is attributed to the anisotropy within the compact and the relationship between sample bending and tensile stresses region. Therefore, three point bending leads to a lower Weibull modulus, attributed to the influence of compact anisotropy. Moreover, in the single-action uniaxially compacted specimen, the use of the classical Weibull analysis leads to a paradox when different Weibull modulus would be found for TS and TRS (deterministically cracked), both depending on the interaction between the distribution of cracks and the density and stress fields. However Weibull theory assumes that failure initiates from a critical defect, whereas many quasi-brittle PM steels fail gradually in bending. This suggests that some care is needed in using the classical Weibull analysis when there is possibility of interaction of defects, or interaction of defects with the stress field gradient.

# 4.3. The ratio of TRS/TS in terms of the Weibull modulus

Much useful information is available through the Weibull analysis. Among the most useful applications are comparisons of strength values and ranges for different stress configurations, Weibull theory predicts a higher strength in bending than in tension. This is because only half of three-point bend specimens is subjected to a tensile stress, and in that portion the stress varies from maximum to zero - as we move from tensile surface mid-span to the neutral axis and to the span extremities, respectively. This investigation also shows higher strength in bending than in tension, with a ratio of similar order of magnitude to that predicted by the Weibull theory. The predicted values follow the observed trends. The present study explored the variation in the TS and UTS. Sintered steel is an inherent random structure having irregularities, such as closed pores, detached and varying ligament sizes, and pore sizes. These abnormalities are even more pronounced at lower porosities, and can affect the mechanical properties that are needed for the various applications of PM steel parts. Thus, three point bending leads to a apparent higher failure stress, attributed to the influence of compact plastic deformation.

A theory was defined for distributed ligaments and cracks very much larger than the ligaments, and in this case, not the crack distribution, but the ligament distribution gives the Weibull modulus. When both the crack lengths and the ligaments sizes are statistically distributed, the expected Weibull modulus will be a weighted average between the modulus of the cracks' distribution and that of the ligaments' distribution, with weight depending on the ratio between the characteristic sizes of cracks and ligaments [26].

Weibull weakest link model, the size/strength relationship can be expressed:

$$\frac{\sigma_{TRS}}{\sigma_{TS}} = \left(\frac{V_{TS}}{V_{TRS}}\right)^{\frac{1}{\overline{m}}}$$
(14)

where *m* is the Weibull modulus,  $\sigma_{TS}$  and  $\sigma_{TRS}$  are the mean strengths of test specimens of type 1 and 2 which may have different sizes and stress distributions, and  $V_{TS}$  and  $V_{TRS}$  are the associated effective volumes of TS and TRS specimen. Weibull modulus was assumed as a weighted average between the TS and TRS modulus. In equation (9) effective areas may be substituted

for the effective volumes for surface flaws. Consequently, if quasi-brittle steel in the form of ISO 2740 specimen, satisfying the conditions of the same shape, size, and square section, is tested at the same strain-rate in two alternative ways:

- in uniaxial tension, with a mean tensile stress at failure sTS;
- in three-point bending, with a mean maximum bending stress at failure of sTRS

depending on the scatter, quantified by the mean Weibull modulus  $\overline{m}$  in the two-parameter analysis (when Weibull moduli for tensile and bend strength should be approximately similar for both testing regimes), the ratio of strengths in terms of the Weibull modulus is:

$$\frac{\sigma_{TRS}}{\sigma_{TS}} = \left\{ 2\left(\overline{m}+1\right)^2 \right\}^{\frac{1}{\overline{m}}}$$
(15)

when strength depends on volume flaws, and

$$\frac{\sigma_{TRS}}{\sigma_{TS}} = \left\{ \frac{4(\overline{m}+1)^2}{\overline{m}+2} \right\}^{\frac{1}{\overline{m}}}$$
(16)

when surface flaws control the strength [27]. An unimodal flaw population that is uniformly distributed throughout the volume and a Weibull two parameter distribution are assumed. However, one major drawback of simple single-action uniaxial compaction is the asymmetrical density gradient and, consequently, an asymmetrical pore structure, which leads to density, porosity and strength gradient also after sintering. Some packing flaws, which are not completely filled during the deformation stage, remain and large density gradients are formed in single-action uniaxial pressing. The "volume" flaws calculation results are generally slightly closer to the measured values TRS/TS than the "surface" flaws calculated  $\sigma_{TRS}/\sigma_{TS}$ .

# 5. Conclusions

1. The strength of quasi-brittle PM steels is a complex parameter which cannot be fully described with single engineering parameter. Therefore, the tensile strength (TS) and transverse rupture strength (TRS) data from 12 batches of PM steel specimens have been analysed by using the 2-p and 3-p Weibull statistics. The Weibull distributions were adopted since they can be deducted

from the statistical distribution of crack initiating flaws (pores and oxide inclusions).

- 2. All predictions generally follow trends in the experimental data, but the 3-p model gives better agreement (except for specimens S+C 1250 TS). It can be assumed that the value of the threshold stress,  $\sigma_u$ , is a good measure of the strength of the material. However, this is still under investigation. It is considered that the 3-p Weibull distribution offers greater possibilities for interpretation. It enables finding results with errors and deleting them, which does not give the 2-p distribution analysis.
- 3. It is shown that the Weibull statistics provides a useful description of the intrinsic statistical description in the fracture stress of quasi-brittle PM steels. The Weibull modulus is an important parameter although it is not a material constant but reflects the shape of the flaw population present in the material.
- 4. Weibull analysis can explain experimental strength data of quasi-brittle sintered steel to some extent, but when there is interaction between pores/cracks, or between pores/ cracks and the gradient of the stress field, the modulus is found to depend significantly on loading (tensile, bending) and geometrical factors, and the Weibull modulus is not a material constant.
- 5. Of particular importance was the observation that the UTS and TRS measurements followed a Weibull probability distribution, with reasonable, but various Weibull modulus. The Weibull analysis of the UTS and TRS data strongly suggests that the large variances in fracture strength data result from a distribution of preexisting defects in the material. These findings justify a damage-mechanics probabilistic approach to studies of sintered steel failure.
- 6. Mechanical properties after sinteraustempering are higher than those attainable with sinterhardening (except TRS after sintering at 1250°C); additionally the sinteraustempered steel does not require any tempering - which causes cost saving. The results show clearly the lower strengths of the sintered and cooled at 10K/min (S+C) materials. They also show that increasing the sintering temperature from 1120 to 1250°C significantly affects TS and TRS.

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TABLE 5

Ratio of TRS to TS calculated from ratio of sTRS to sTS by using formulae (15) and (16)

| Sintering<br>temperature | Specimens designation | Number of specimens, N | TRS/TS<br>measured | σ <sub>TRS</sub> /σ <sub>TS</sub> ; "surface"<br>flaws | $\sigma_{TRS}/\sigma_{TS}$ ; "volume" flaws |
|--------------------------|-----------------------|------------------------|--------------------|--|---|
| 1120°C                   | SAT 500               | 25                     | 1.76               | 1.33   | 1.63  |
|                          | S+H                   | 24                     | 1.81               | 1.23   | 1.38  |
|                          | S+C                   | 24                     | 1.86               | 1.36   | 1.70  |
|                          | SAT 500               | 25                     | 1.71               | 1.33   | 1.63  |
| 1250°C                   | S+H                   | 25                     | 1.86               | 1.27   | 1.45  |
|                          | S+C                   | 24                     | 1.86               | 1.31   | 1.56  |

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