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Evaluation of maximum non-metallic inclusion sizes in engineering steels by fitting a generalized extreme value distribution based on vectors of largest observations

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ABSTRACT

In the analysis of maximum sizes of large non-metallic inclusions in steels, the common extreme value analysis is only based on a sample of largest observations in control areas on polished planes, respectively. From a simulation study it is observed that this univariate set-up may lead to a high proportion of misspecifications of the true extreme value distribution. Due to their different tail behavior, a falsely determined extreme value distribution will lead to erroneous assertions about quantiles, which serve as characteristic quantities. A multivariate extreme value analysis incorporating data of the r largest observations in every control area will work satisfactorily and should be preferred. The effects of different choices of r are also illustrated by means of a real data set of oxide inclusions.

1. Introduction

Non-metallic inclusions are compound materials of metals (e.g., iron, manganese, silicon, aluminum) with non-metals (especially oxygen and sulfur) embedded inside steel during the manufacturing process.

The source of inclusions can be exogenous (embedding of particles) when the metal passes through the lining of furnaces, ladles or casting devices; these inclusions are very rare in modern steels. Endogenous inclusions are formed by precipitation inside the melt or the solidified material due to decreasing solubility with falling temperature. This paper deals with endogenous non-metallic inclusions.

Very small inclusions with a size of a few nm develop as precipitates during solid steel processing. Significant larger inclusions with sizes of a few μm derive from precipitation during deoxidation and casting. Thus large primary precipitates from the melt, medium size secondary precipitates from the high temperature fcc phase of steel and small tertiary precipitates from the low temperature bcc phase can be distinguished. This paper deals with large size primary inclusions.

By chemical content non-metallic inclusions can be divided into oxides, sulfides, nitrides and phosphides. The majority of inclusions in steels are sulfides and oxides. Since the latter are most harmful for the properties of steels this paper deals with oxides.

Non-metallic inclusions are a matter of concern with respect to several properties of steel, such as fatigue, formability, toughness, machinability and corrosion resistance.

Especially μm scale inclusions are considered as nuclei for pores and crack initiation and by this being responsible for the damage development during mechanical testing. It is therefore of prime importance to develop steels with highest purity; that is freedom of large non-metallic inclusions and a small homogeneous distribution of the unavoidable rest. The statistical distribution of maximum inclusion sizes are relevant for the prediction of the material behavior especially when cyclic loading is applied or when highest toughness requirements have to be met. Material's damage corresponds to its microscopic behavior and is defined as an irreversible degradation on a given length scale. This length scale varies from a few microns to a dozen of microns depending on the material, microstructure and loading conditions (cf. [1]). This paper deals with inclusions on the μm scale that are considered critical for cyclic loadings.

The inclusion distribution can be investigated by various metallographic or non-destructive methods like ultra sonic investigations. By metallographic means the size, shape and arrangement can be determined quantitatively in the investigated control area of the sample. This paper deals how experimental input data can

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be evaluated by statistical methods in order to describe the distribution function of maximum particle sizes and to predict the probability of large diameter inclusions.

2. Experimental materials and methods

2.1. Experimental materials

The experiments have been carried out using the precipitation hardening ferritic–pearlitic steel 38MnVS6 according to DIN EN 10267 [2]. The chemical composition is provided in Table 1. The relatively high sulfur content of 0.047 has been added in order to improve the machinability, the alloying element V is used for precipitation hardening during cooling from hot-working temperatures.

The steel has been melted in an Electric Arc Furnace and continuously casted to a billet with the cross section of $240 \times 240\ \text{mm}^2$ followed by hot rolling to a round bar of diameter of 61 mm. From these bars samples were taken according to the standard DIN EN 10247 [3], Fig. 1. The analysis of nonmetallic inclusions was then performed following the ASTM-standard E2283-08 [4]. To give more details, a number of plane surfaces of size $200\ \text{mm}^2$ have been polished. On these polished planes, in total 60 control areas, each of size $0.76786\ \text{mm}^2$, have been chosen.

Commonly, only the largest inclusion is determined in each control area. In our data set, all inclusions were incorporated having a maximum diameter of at least 5 µm; for each inclusion, the so-called $\sqrt{\ }$area-parameter has been recorded, which is defined to be the square root of the projected area of an inclusion (cf. [5,6]).

In our particle analysis, two major inclusion types were differentiated, namely globular oxides and sulfides. Exemplarily, light optical microscope pictures of these two inclusion types are shown in Fig. 2.

In our study, we are concerned with oxides, since these inclusions are seen to be more critical regarding fatigue life of components. All particles with a ratio of maximum and minimum diameter smaller than 3 have been considered globular oxides. This assumption is feasible, as the steel is highly deformed so that the soft sulfides should be stretched to a larger extend than the hard oxides. In our data set obtained by the described procedure, the maximum number of inclusions per control area is given by 81.

2.2. The control area maxima method

In a series of papers Murakami and his co-workers established a method, frequently termed the control area maxima approach, to estimate the size of the largest two-dimensional inclusion that can be found in a certain reference area that is larger than the control areas used for measuring; see Murakami [5], Murakami et al. [7] and the references therein. Given the maximum inclusion size of each control area, Murakami's work is based on the Gumbel family of distributions, having distribution functions

$$\exp\left(-\exp\left(-\frac{x-\mu}{\sigma}\right)\right), \quad x \in \mathbb{R}, \tag{1}$$

with location parameter $\mu \in \mathbb{R}$ and scale parameter $\sigma > 0$. The fitting of a Gumbel distribution (in terms of the maximum likelihood (ML) principle) to the maximum inclusion sizes of $N \in \mathbb{N}$ control areas has found entrance in technical recommendations: see ESIS

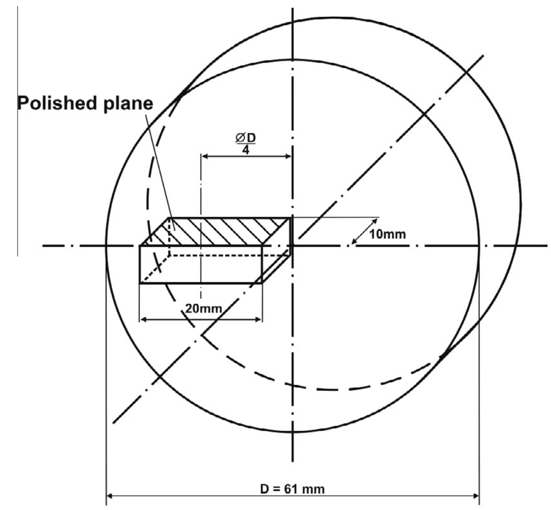

Fig. 1. Sample preparation from the steel bar according to DIN EN 10247 [3].

P11-02 by Anderson et al. [6] and E2283-08 by ASTM International [4]. Having once numerically determined ML estimates $\widehat{\mu}$ and $\widehat{\sigma}$ of the unknown distribution parameters $\mu \in \mathbb{R}$ and $\sigma > 0$, the p-quantile estimate

$$\widehat{x}_p = \widehat{\mu} - \widehat{\sigma}\ln(-\ln p)$$

is recommended to be calculated, with $p \in (0, 1)$, such as $p = 0.999$ (cf. [4]). As an issue of prediction or rather extrapolation, such a p-quantile is called the characteristic size of the largest inclusion with respect to the return period $T = 1/(1-p)$, being the size that is expected to be exceeded exactly once in a reference area that is T times larger than the control areas, or, in other words, being the size of an inclusion in a polished plane that is expected to be exceeded by exactly one maximum inclusion in T control areas (see, e.g., [6]).

When considering limit distributions of normalized maxima in extreme value theory, Gumbel distributions as in (1) as well as two more families of distributions appear, namely Fréchet and reversed Weibull families. Thus, the question arises whether it is always reasonable to restrict to Gumbel distributions, and then to look for the best fit based on observed control area maxima. E.g., with estimating quantiles in view, a prefixed, but non-appropriate choice of the family of extreme value distributions will have serious consequences in practice (see Sections 3 and 4). The use of a Gumbel distribution has been frequently reasoned by the argument that some measurements have shown that the distribution of inclusion sizes in steels is nearly described by a log-normal, exponential or Weibull distribution (cf. 6,8,9]), which belong to the Gumbel domain of attraction, i.e., normalized maxima from samples from one of the above distributions are theoretically seen to converge to a Gumbel distribution. Rather than adopting one predetermined extreme value family, it is more common in applied statistics (cf. Coles 10,11]) to consider the generalized extreme value (GEV) family of distributions, with distribution functions

$$G_k(x) = \exp\left(-\left(1 - k\,\frac{x-\mu}{\sigma}\right)^{1/k}\right), \quad 1 - k\,\frac{x-\mu}{\sigma} > 0, \tag{2}$$

which contains Gumbel (w.r.t. $k \to 0$), Fréchet and reversed Weibull distributions as particular cases. In addition to a location parameter

Table 1
Chemical composition (weight-%) of the investigated steel (38MnVS6).

Steel	Composition (wt.-%) C	Si	Mn	P	S	Cr	Ni	Al	Cu	N	Nb	Ti	V
38MnVS6	0.39	0.59	1.41	0.016	0.047	0.19	0.08	0.011	0.09	0.01	0.002	0.002	0.10