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Procedia Materials Science (2014) 000–000

Procedia Materials Science

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20th European Conference on Fracture (ECF20)

Estimation of single-grain properties of steel through inverse-engineering of microindentation experiments: Uniqueness of the solution

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Abstract

Cleavage fracture of steel is governed by micromechanical mechanisms, but it is modelled with simple continuum-level models. These models are starting to reach the limits of their applicability, so there is interest to look deeper into micromechanical modelling of fracture processes. In order to model fracture on the microstructural level, a plasticity model of the grains themselves is needed. In prior studies, a method of finding the single-crystal properties by an inverse engineering approach has been described. By this method, a single crystal is indented, and the material input to a FE model of the experiment is iteratively changed until agreement is achieved. These prior studies have shown that methods like this provide ambiguous results. In this paper, the uniqueness of the solution is assessed by evaluating the difference between experiments and simulations for a variety of material input parameters. The results show that with a power law hardening relationship, martensite produces two unique minima (at n=0 and 0.1). This may represent the Lüder's plateau and the subsequent hardening, respectively. The ferrite material featured a minimum that was flat, but the flatness was over a very short range of hardening exponents.

Keywords: Microindentation; micromechanics; inverse engineering; single-grain plasticity

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1. Introduction

Prevention of cleavage fracture remains a major concern in most industrial applications of steel structures. The relationship between macroscopic loading and material behavior at the microstructure level is one of the key aspects necessary to give a better understanding of cleavage fracture. Steel manufacturers can control certain microstructural features (e.g. prior austenite grain size), but due to a lack of knowledge about how these features affect the fracture mechanical material behavior, they don't know how some features of the microstructure should be modified in order to obtain better performance against cleavage fracture. Therefore, there is an increasing interest in micromechanical modeling of the cleavage fracture process. The plastic material behavior of individual grains is needed in order to model microstructural fracture.

Indentation tests have been used to determine mechanical properties of materials. Many researchers, e.g. Venkatesh et al. (2000), Dao et al. (2001), Bucaille et al. (2003), Lee et al. (2005), have been using indentation data to extract elastic-plastic material properties based on inverse engineering. Several studies (e.g. Chen et al. 2007, Ma et al. 2012) have shown that it is difficult to obtain an unique set of material properties from the load-displacement curve of one single indentation test. Therefore, some researchers, e.g. Bucaille et al. (2003), Luo and Lin (2007), have investigated dual sharp indentation in order to determine an unique solution. This method uses different indenter geometries to obtain the load-displacement curves. However, the sensitivity and uniqueness of the determined material properties are still issues to deal with.

In this paper, a method is described to determine plasticity data from a single indentation test, based on the optimization approach of Kang et al. (2012), in which a microindentation test is iteratively simulated in order to identify the relevant plasticity parameters. However, unlike Kang et al. (2012), the current study assesses the impact of the plasticity model on the error function in order to gain insight into the uniqueness of the solution.

Nomenclature

σ	stress
ε	strain
K	hardening coefficient
n	hardening exponent
error	area difference between experimental and simulation load displacement curve
F_{test}	force of indentation test at given displacement
F_{FEA}	force of FE simulation at given displacement
Δ	displacement
Δ_{max}	displacement at maximum force
Δ_0	displacement when unloading force returns to zero

2. Microindentation experiments

A test sample of a S690 structural grade steel was prepared, and the surface of the sample was polished to create a smooth surface. Indentation measurements were performed on an universal nanomechanical tester (UNAT) equipped with a Berkovich tip, which is a three-sided pyramidal indenter. The indenter has a tip radius of 1 μm, and an indentation load of 100 mN was used. Scanning electron microscope (SEM) images of the microstructure were made prior the indentation measurements, and indentation measurements on single grains at ten different locations of the test sample were performed for each phase. From the SEM images, the average prior austenite grain size was determined according to ASTM E112-10, and it was found to be approximately 20 μm. Typical raw force versus displacement result for both phases are shown in Figure 1.

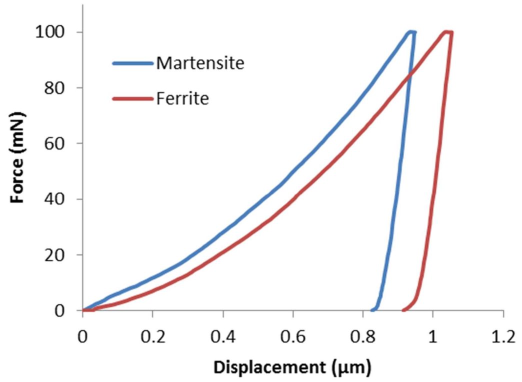

Fig. 1. Force displacement curves from indentation measurements.

3. Analysis

3.1 Finite Element model

Simulations of the microindentation experiments were performed to determine the plasticity properties of the two main microstructural components, martensite and ferrite. The finite element analysis of the indentation measurements are based on a 2D axisymmetric model using Abaqus/CAE 6.12-1. In order to define an axisymmetric model of the Berkovich indenter, a rigid conical surface with half-apex angle of 70.3° and a tip radius of 1 μm was assumed. Linchini et al. (1998), have verified that the 2D axisymmetric approach can be used to simulate Berkovich indentation experiments. The mesh of the steel test specimen should be small enough to reduce the computation time but large enough to minimize the effects of the boundary conditions on the indented region. Therefore, the test specimen is modelled as a square of 50x50 μm. The region of interest is the contact area of the indenter with the specimen, and that is very small compared to the size of the mesh. High stresses and large deformations are expected immediately below the indenter tip. For accurate results, a fine mesh is used near the contact area, and a gradually coarser mesh is used further away from the contact area, as shown in Figure 2. The region below the indenter tip is taken as a small square of 4x4 μm and meshed with 60x60 four-node axisymmetric quadrilateral continuum elements (CAX4) of equal size. Reduced integration elements (CAX4R) are used for the elements outside this small square to reduce the computational time.

(a) (b)

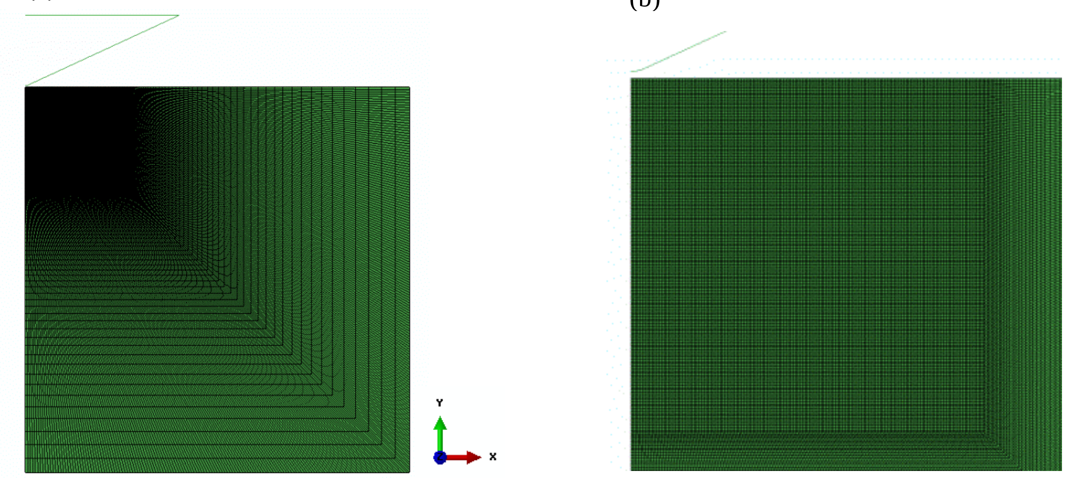

Fig. 2. (a) Mesh of the finite element model; (b) Zoomed in on region below indenter tip.

All of the nodes at the bottom of the specimen are fixed to prevent them of moving in x and y direction. The axis of symmetry is the y-axis, and the nodes at this axis can only move in downwards direction. The contact between the tip and the specimen is modelled as a surface-to-surface contact with the tip taken as the master surface and the top surface of the test specimen as the slave surface. The friction coefficient between the tip and the specimen surface is assumed to be zero. The indentation measurement is simulated by two steps: loading and unloading. During the loading step, a displacement in downwards y direction is imposed at the tip equal to the maximum penetration depth measured during the indentation experiments. The tip returns to the initial position during the unloading step.

3.2 Optimization procedure

The material behavior in the FE simulations is described with a power law strain hardening curve. For power law materials, the stress-strain (σ-ε) relationship is assumed to be:

$$\sigma = K\varepsilon^n \tag{1}$$

where K is the hardening coefficient and n the hardening exponent. Such a model has been used in several studies, e.g. Dao et al. (2001), Bucaille (2003), Luo and Lin (2007). The material was assumed to be isotropic with a von Mises yield surface and associated flow rule. An automated procedure of several steps for determining the hardening properties of martensite and ferrite is developed within MATLAB. Initial guess values for the parameters K and n are provided to describe the stress-strain characteristics of the indented material in the FE model. The finite element simulation of the indentation experiment is performed, and a load displacement curve is extracted from the output file. The FE load versus displacement curve is compared to an indentation test curve. The area difference is calculated according to the following equation:

$$error = \int_0^{\Delta_{max}} |F_{test} - F_{FEA}| d\Delta + \int_{\Delta_{max}}^{\Delta_0} |F_{test} - F_{FEA}| d\Delta \tag{2}$$

where *error* is the value used for comparison, F_{test} is the force of the test at a given displacement, F_{FEA} is the force of the FEA at a given displacement, Δ is the displacement, Δ_{max} is the displacement at the maximum force, and Δ_0 is the displacement when the unloading force returns to zero. The parameters K and n are iteratively changed until Equation (2) is minimized. This iterative process is automated through the MATLAB fmincon command. The flow chart of this procedure is schematically shown in Figure 3.

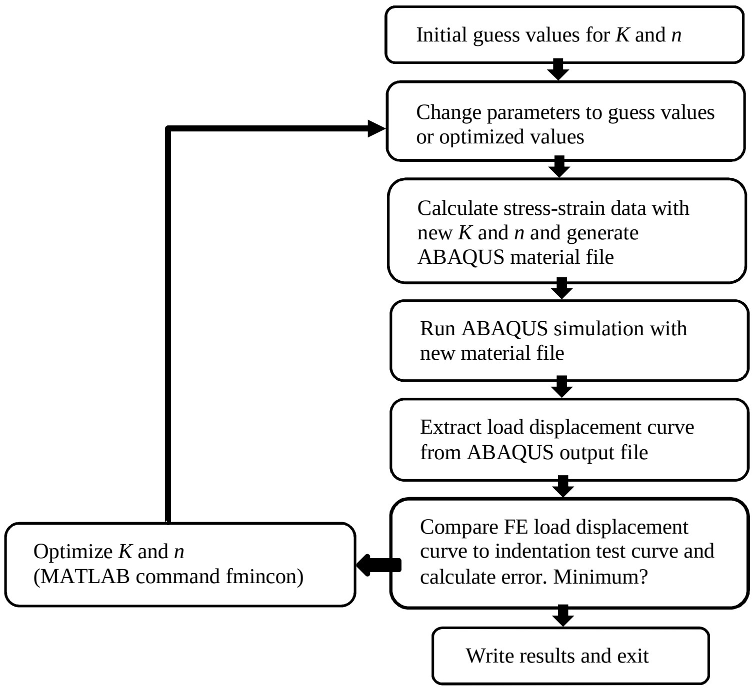

Fig. 3. Flow chart of optimization procedure.

The first approach was to optimize both parameters *K* and *n* simultaneously. The numerical minimizer that was used in MATLAB delivered inconsistent results with the two-parameter optimization. Therefore, a new approach was followed whereby several values of *n* were specified, and MATLAB was allowed to optimize the *K* to find the lowest *error* for each of the specified values of *n*. The number of iterations was minimized by using an initial guess of the *K* parameter that delivered the same specific plastic energy as the first optimized value, which is illustrated in the figure below.

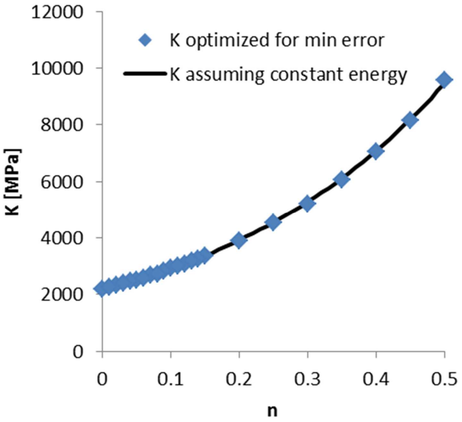

Fig. 4. Constant specific energy curve.

This is done for a sequence of *n* values in the range of 0-0.5. The relative error (defined as the difference between the error for a particular *n* and the minimum *error*), is calculated for both martensite and ferrite. The results are presented in Figure 5. It can be seen that martensite has two unique minima, *n=0* and *n=0.1*. Ferrite has a plateau of minima between *n=0.415* and *n=0.417*.

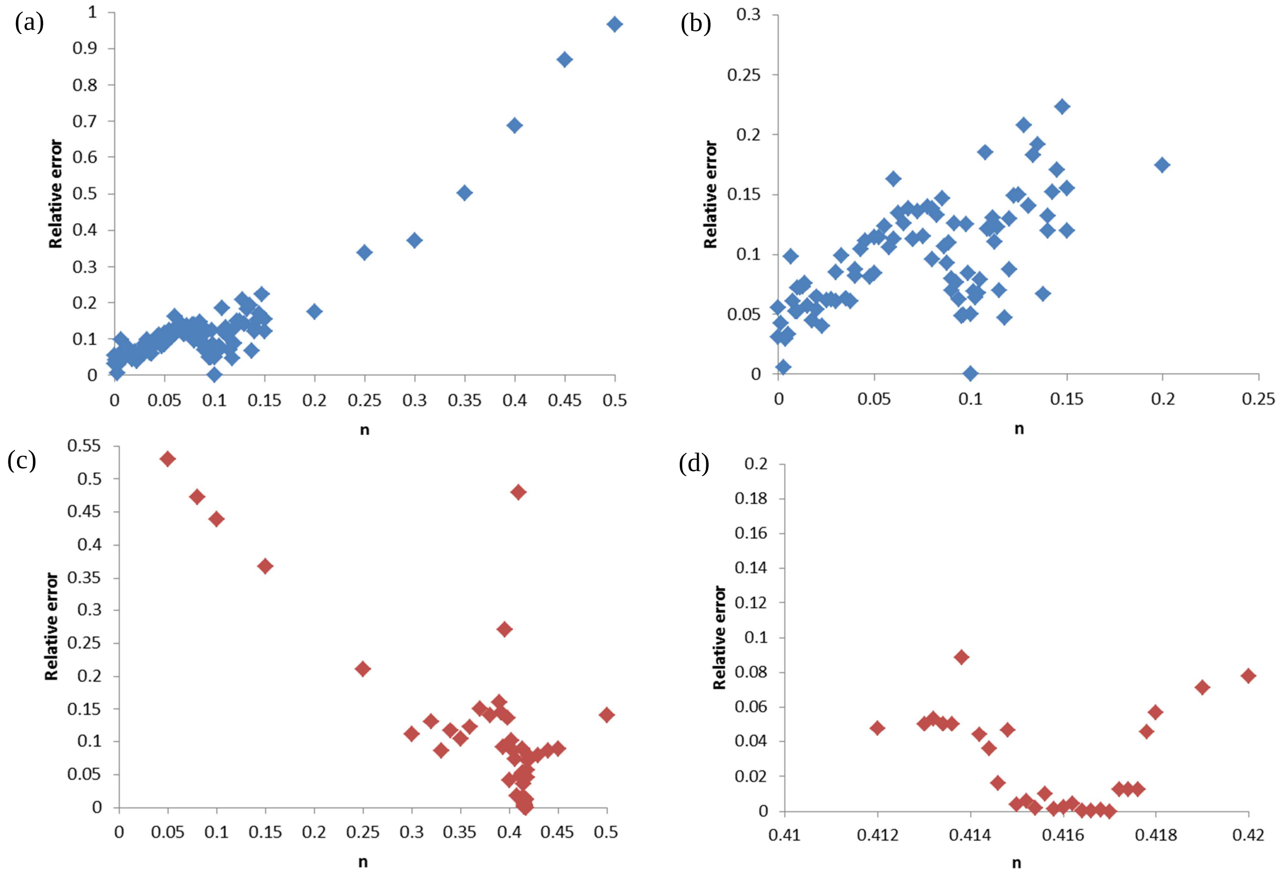

Fig. 5. (a) Relative error for martensite; (b) Zoomed in on minima; (c) Relative error for ferrite; (d) Zoomed in on minima.

A possible explanation for the two unique minima found of *n* for martensite, is the possible presence of the Lüder's band, which can give a flat stress versus strain curve followed by normal hardening. The stress-strain curve of the material in the Lüder's band would have a hardening exponent $n=0$. After the Lüder's band, strain hardening will follow, and thus giving the minimum of $n=0.1$. The error between simulation and experiment is approximately 2.9% and 1.5% for martensite and ferrite respectively. Furthermore, the hardening parameters determined for martensite by this optimization approach, are in the same order of the hardening parameters found in literature, e.g. Al-Abbassi and Nemes (2003).

4. Conclusion

Indentation measurements are commonly used to analyze elastic-plastic material properties where standard tension tests are not practical. Finite element modeling is used to extract the plasticity properties by fitting the experimental data. An important issue in this approach is the uniqueness of the obtained material properties. The study presented in this paper shows that, assuming a power law hardening relationship, unique hardening parameters can be estimated from a load displacement curve measured from one single indentation test. Therefore, a practical procedure based on the optimization approach is demonstrated to determine accurate plastic material properties of steel with indentation experiments using a Berkovich indenter. In order to gain insight into the uniqueness of the solution, the impact of different stress versus strain hardening relationships on the quantitative error function is evaluated. The results show that it is possible to obtain an unique set of hardening parameters with good accuracy between experiment and simulation. Further research is required to improve the accuracy and uniqueness of the hardening parameters. Further research is also necessary to confirm that the two minima associated with the martensite do indeed correspond to Lüder's bands and subsequent hardening.

Acknowledgements

The funding of the Enabling Technology Program (ETP) of TNO through the CMS project is gratefully acknowledged.

References

ASTM E112 – 10, 2010. Standard Test Methods for Determining Average Grain Size, ASTM International.

Al-Abbasi, F.M., Nemes, J.A., 2003. Micromechanical modeling of dual phase steels. International Journal of Mechanical Sciences 45, 1449-1465.

Bucaille, J.L., Stauss, S., Felder, E., Michler, J., 2003. Determination of plastic properties of metals by instrumented indentation using different sharp indenters. Acta Materialia. 51, 1663-1678.

Chen, X., Ogasawara, N., Zhao, M., Chiba, N., 2007. On the uniqueness of measuring elastoplastic properties from indentation: The indistinguishable mystical materials. Journal of the Mechanics and Physics of Solids 55, 1618-1660.

Dao, M., Chollacoop, N., van Vliet, K.J., Venkatesh, T.A., Suresh, S., 2001. Computational modeling of the forward and reverse problems in instrumented sharp indentation. Acta Materialia. 49, 3899-3918.

Kang, J.J., Becker, A.A., Sun, W., 2012. Determining elastic-plastic properties from indentation data obtained from finite element simulations and experimental results. International Journal of Mechanical Sciences 62, 34-46.

Lee, H., Lee, J.H., Pharr, G.M., 2005. A numerical approach to spherical indentation techniques for material property evaluation. Journal of the Mechanics and Physics of Solids 53, 2037-2069.

Lichinchi, M., Lenardi, C., Haupt, J., Vitali, R., 1998. Simulation of Berkovich nanoindentation experiments on thin films using finite element method. Thin Solid Films 312, 240-248.

Luo, J., Lin, J., 2007. A study on the determination of plastic properties of metals by instrumented indentation using two sharp indenters. International Journal of Solids and Structures 44, 5803-5817.

Ma, Z.S., Zhou, Y.C., Long, S.G., Zhong, X.L., Lu, C., 2012. Characterization of stress-strain relationships of elastoplastic materials: An improved method with conical and pyramidal indenters. Mechanics of Materials 54, 113-123.

Venkatesh, T.A., van Vliet, K.J., Giannakopoulos, A.E., Suresh, S., 2000. Determination of elasto-plastic properties by instrumented sharp indentation: guidelines for property extraction. Scripta mater. 42, 833-839.