

## Characterization of reactor vessel steels in the brittle-ductile transition region

T. Linse<sup>1,a</sup>, M. Kuna<sup>1,b</sup>

<sup>1</sup>TU Bergakademie Freiberg, Institute of Mechanics and Fluid Dynamics, Lampadiusstrasse 4, Freiberg, 09596 Germany

<sup>a</sup>thomas.linse@imfd.tu-freiberg.de, <sup>b</sup>meinhard.kuna@imfd.tu-freiberg.de

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**Abstract.** In the brittle-ductile transition region, ferritic steels fail due to transcrystalline or intercrystalline cleavage fracture initiating at flaws resulting from microplastic deformations. The large scatter of fracture mechanical properties in the transition region requires the statistical analysis of large amounts of tests while the available material for testing is usually limited to small volumes, especially for irradiated material. Two reactor vessel steels are investigated using the Small-Punch-Test, a miniaturized test method. Samples are taken from remnants of already tested Charpy-specimens. The experiments are conducted with non-irradiated and irradiated specimens of both steels at different temperatures, thereby covering the brittle and brittle-ductile transition region. An identification method using Neural Networks is applied to analyze the load-displacement-curve of the Small-Punch-Test regarding its information about the material behavior. The Beremin local approach model is used to quantify the probability of cleavage fracture for a certain load state. For one non-irradiated material, the determined material properties are used to estimate critical fracture toughness values by simulating fracture mechanical tests.

### Introduction

Minimal invasive sampling and testing of miniaturized specimens are the predestined choice for the characterization of reactor vessel steels. As irradiation yields to the embrittlement of the steels, a sufficient large amount of tests is needed for the description of the brittle and transition region, which changes during service. To warrant the high safety standards of existing nuclear power plants, the quantification of the effects of irradiation is essential.

The small punch test (SPT) [1]-[8] is a suitable device for the surveillance of critical parts of nuclear power plants, as its specimens require only tiny volumes of material, which makes a quasi non-destructive minimal invasive sampling possible. Furthermore, the specimens can be manufactured from remnants of already tested Charpy specimen, which makes the SPT a valuable completion to standard testing methods. The test itself can be considered as miniaturized deep drawing process until failure. A quadratic specimen, supported by a die, is loaded centrally and deformed vertically by a punch.

The essential measured output of the SPT, the load displacement curve (LDC), contains valuable information about the elastic, hardening and fracture mechanical properties of the tested material. It can be divided into five characteristic parts (Figure 1). Additional information about the type of failure of the specimen can be obtained by analyzing the resulting fracture surfaces.

Cleavage fracture, the dominant type of failure in the brittle and transition region, typically initiates at microscopic flaws that nucleate at carbides or other hard non-metallic inclusions in ferritic steels as a result of microplastic deformations. As the statistical distribution of these microcracks causes a large scatter of fracture mechanical properties, a probabilistic model is required to describe the failure in the brittle or transition region. The local approach model by Beremin [9][10] is able to quantify the probability of cleavage fracture for a certain load case based on the weakest link theory.

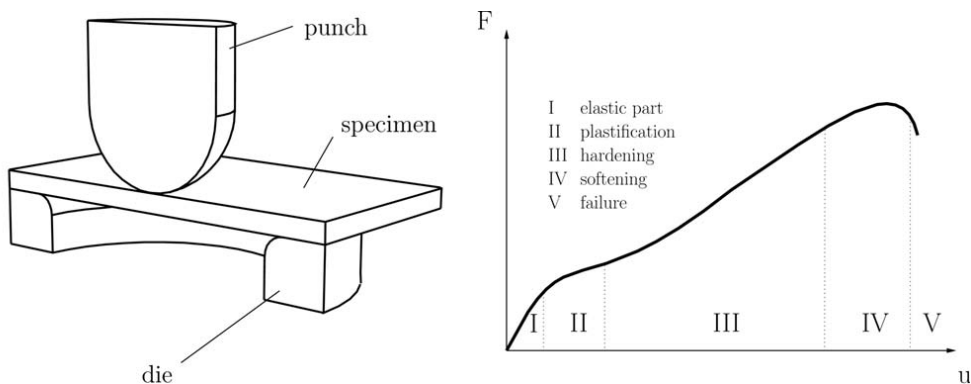


Figure 1: SPT principle (left) and characteristic load displacement curve (right)

In the present paper we characterize material properties of two ferritic steels by means of the SPT, thereby investigating the influence of irradiation and temperature. Neural networks are used to solve the inverse problem in order to identify mechanical properties from measured LDCs. The identified values are compared with results from standard test methods.

### Experimental Work

**Investigated Materials.** Two ferritic reactor vessel steels, A533 Cl. 1 (IAEA JRQ) and A508 Cl. 3 (IAEA JFL) were investigated using the SPT. Non-irradiated and irradiated specimens of both materials were tested at different temperatures, thereby covering brittle (B), transition (T) and ductile (D) regions of the steels (see Table 1 and Table 2).

Table 1: Test program material JFL

Temperature	-185°C	-175°C	-155°C	-135°C	-125°C	-105°C	-80°C	+22°C
Irradiation [10 <sup>18</sup> n/cm <sup>2</sup> ]								
non-irradiated	B	B	B	T	T	T	D	D
RH6 (7,15)				B			T	X
RH7 cf (51,21)				B			T	X
RH7 cn (86,69)				B			T	X

Table 2: Test program material JRQ

Temperature	-150°C	-140°C	-120°C	-100°C	-70°C	-50°C	-30°C	+22°C	+70°C
Irradiation [10 <sup>18</sup> n/cm <sup>2</sup> ]									
non-irradiated	B	B	T	T	D	D	D	D	
RH6 (7,14)			B		T		D		
RH7 cf (54,85)					B		T	D	
RH7 cn (98,18)							B	T	D

**SPT Setup.** The SPT device is built in a modular way to enable the handling with manipulators in a hot cell [3]. Using a cooling system based on liquid nitrogen, or alternatively a heating device, testing is possible at temperatures between -185°C and +150°C. The SPT device is installed into a standard testing machine with a 10 kN load frame.

The test geometry (punch radius  $R=2.0$  mm, edge radius  $r=0.5$  mm, inner diameter of the die  $d=6.8$  mm) was chosen to yield as high as possible first principal stresses with small stress gradients in a large continuous volume inside the specimen. The specimen is not clamped using a second upper die, which limits the possible area of failure.

**Specimen Sampling.** The quadratic SPT specimens (10x10 mm<sup>2</sup>, thickness 0.5 mm) are machined from remnants of already tested Charpy specimens using a wire electro-discharge machine. The compression side of the specimens results from a separating cut followed by finishing cuts, while the tension side is additionally ground using P1200 (FEPA) abrasive paper. This preparation methods produces roughness averages of  $R_a=2 \mu\text{m}$  (compression side) and  $R_a=0.04 \mu\text{m}$  (tension side), the thickness of the specimens lies within a tolerance of [0; +5  $\mu\text{m}$ ].

**SP Testing.** The tests are driven displacement-controlled at a constant punch velocity of 0.5 mm/min. First, specimens of non-irradiated material were tested to verify the identification methods and to investigate the influence of the test temperature on the LDCs and the failure behavior. Second, specimens of both materials each at three different irradiation levels were tested at different temperatures to study the influence of irradiation. All together, over 600 SP tests were conducted, thereof 420 using irradiated material.

### Identification of Material Properties

The inverse problem of finding material properties from measured LDCs is solved using neural networks (NNs) that have been trained using a database consisting of material parameters and the corresponding LDCs calculated by FEM. An optimization algorithm minimizes the difference of the measured LDC and the output of the NNs, thus identifying the mechanical properties.

**Constitutive Behavior.** In the beginning, a piecewise linear function [3] with four parameters was used for the yield curves. Now, a better approximation was found using a modified Voce function [11]

$$\sigma_F^V(\varepsilon_{pl}) = \begin{cases} \sigma_1 & 0 \leq \varepsilon_{pl} \leq \varepsilon_L \\ \sigma_1 + \sigma_2(\varepsilon_{pl} - \varepsilon_L) + (\sigma_1 - \sigma_2 - \sigma_3)(1 - e^{-k(\varepsilon_{pl} - \varepsilon_L)}) & \varepsilon_L \leq \varepsilon_{pl} \leq 1 \end{cases} \quad (1)$$

consisting of five degrees of freedom. It accounts for the Lüders strain in a simplified way using the parameter  $\varepsilon_L$ .

**Numerical Simulations.** To build a database consisting of material properties and the associated LDCs of the SPT, the SPT is simulated using an axisymmetric FEM model. Die and punch are modeled as rigid bodies, whereas the elastic-plastic material behavior of the specimen is accounted for by using the von Mises yield potential and the associated flow rule. The contact between specimen, punch and die is modeled with a constant friction coefficient  $\mu=0.2$ .

To create a database for the training of NNs, the five parameters of the yield curve approximation were varied systematically and the LDC of the SPT was calculated for each parameter set.

**Neural Networks.** Single feed forward NNs consisting of three layers, each containing neurons with sigmoid activation functions, were trained to approximate the punch force for a specific punch displacement as a function of the constitutive parameters. The Stuttgart Neural Network Simulator [12][13] was used to create and train the NNs. Overtraining and thus the loss of generalization was controlled by splitting the database into training and validation sets.

**Optimization Algorithm.** The search for the optimal parameter set that gives a minimal difference

$$E = \sqrt{\frac{1}{N} \sum_{n=1}^N \left( \frac{\tilde{F}_{SPT}(u_n) - \tilde{F}_{NNn}(x)}{\tilde{F}_{SPT}(u_n)} \right)^2} \rightarrow \text{Min} \quad (2)$$

between the measured LDC and the output of the NNs is done using the simulated annealing algorithm [14][15]. This global optimization algorithm does not use derivatives of the objective function and allows uphill movements under the control of a probabilistic criterion, which minimizes the probability to get stuck in local minima and the dependency from a good starting point. However, the algorithm needs a larger number of function evaluations compared with other algorithms to find the global minimum.

**Calculation of Weibull Parameters.** The cumulative failure probability due to brittle fracture is modeled using the two-parameter Weibull distribution described by Beremin [9][10]. The Beremin model assumes that cleavage fracture initiates at microscopic cracks that nucleate at hard inclusion in the presence of plastic deformations. The probability density function of the microcracks (length  $l_0$ ) is supposed to follow an inverse power law  $f(l_0) = \alpha/l_0^\beta$ . Following the weakest-link theory, the probability of failure for a specific load state is then given by

$$P_f\{\sigma_W(L)\} = 1 - \exp\left\{-\left(\frac{\sigma_W(L)}{\sigma_u}\right)^m\right\} \quad (3)$$

To determine the two Weibull parameters (module  $m$  and reference stress  $\sigma_u$ ), the stress distributions inside the SPT specimens that failed due to cleavage fracture are calculated using FEM. This is done iteratively, thereby correcting  $m$  and  $\sigma_u$  with the maximum likelihood method until the calculated probability fits the experimental distribution [16].

**Prediction of Fracture Mechanical Properties.** The fracture toughness in the brittle and transition region is estimated by simulating tests with SENB specimen using FEM and the identified hardening parameters. Analyzing the stress distribution inside the specimen and calculating the Weibull stress with the determined Weibull parameters  $m$  and  $\sigma_u$ , critical fracture toughnesses  $K_{Ic}$  are calculated from  $J$ -integral values as the Weibull stress reaches 5%, 50% and 95% probability of cleavage fracture.

## Application and results

**Identified Hardening Parameters.** The explained identification strategy was applied to determine the five yield curve parameters from the LDCs of SPTs carried out for the two materials, both non-irradiated and irradiated, at different temperatures. Figure 2 and Figure 3 show normalized initial yield stresses compared with results from tensile tests (TT). The identified values agree well with measured data.

**Determined Weibull Parameters.** Weibull parameters were determined for those temperatures and irradiation levels, respectively, for which the tested specimens failed in a brittle manner. Using the maximum likelihood method, unreasonable high Weibull modules were obtained for some test series. Therefore, it was decided to set the Weibull module to a constant value of  $m=22$ . Figure 4 and Figure 5 show the determined Weibull plots for both materials. The obtained values show dependencies from both, test temperature and irradiation level, whereas the influence of irradiation dominates for the material JRQ.

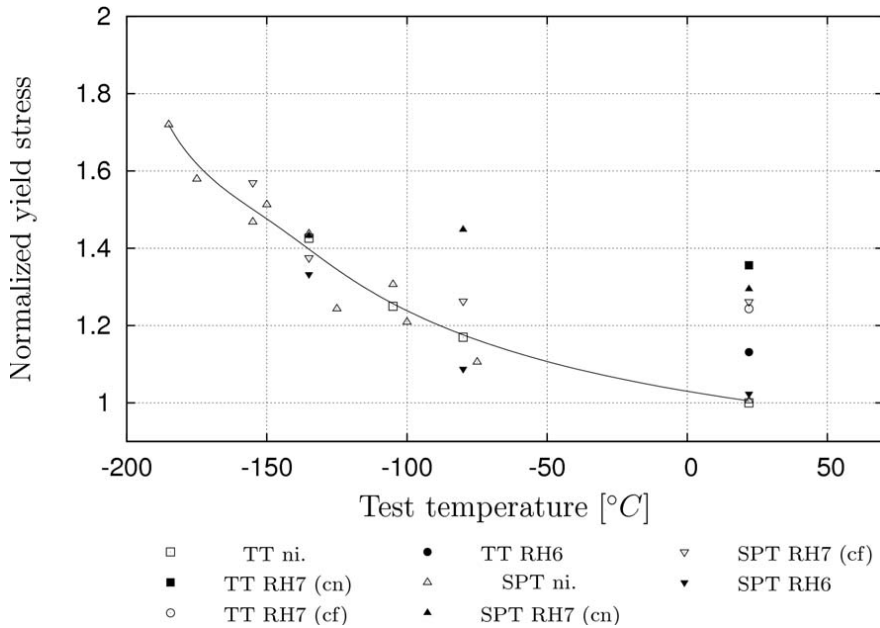


Figure 2: Normalized identified initial yield stresses JFL

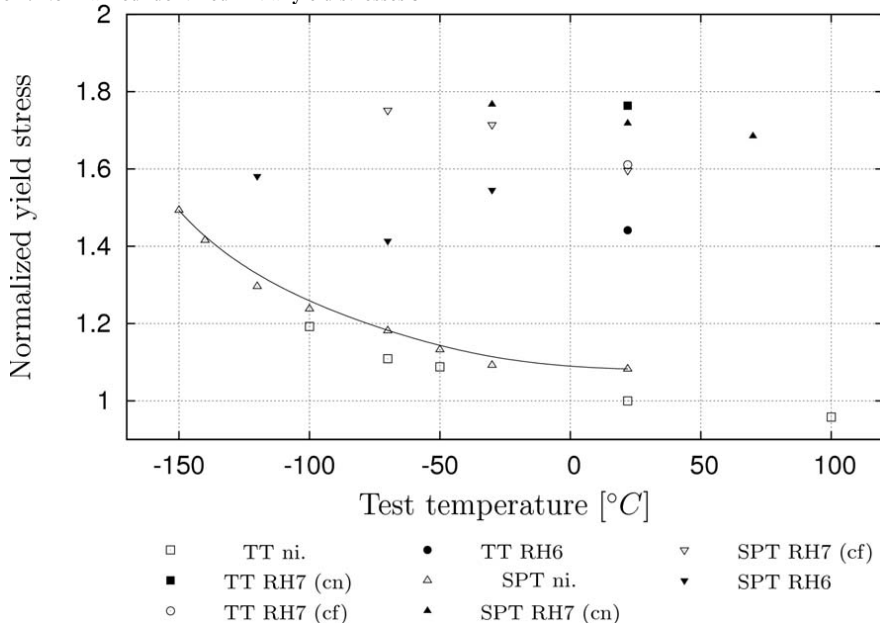


Figure 3: Normalized identified initial yield stresses JRQ

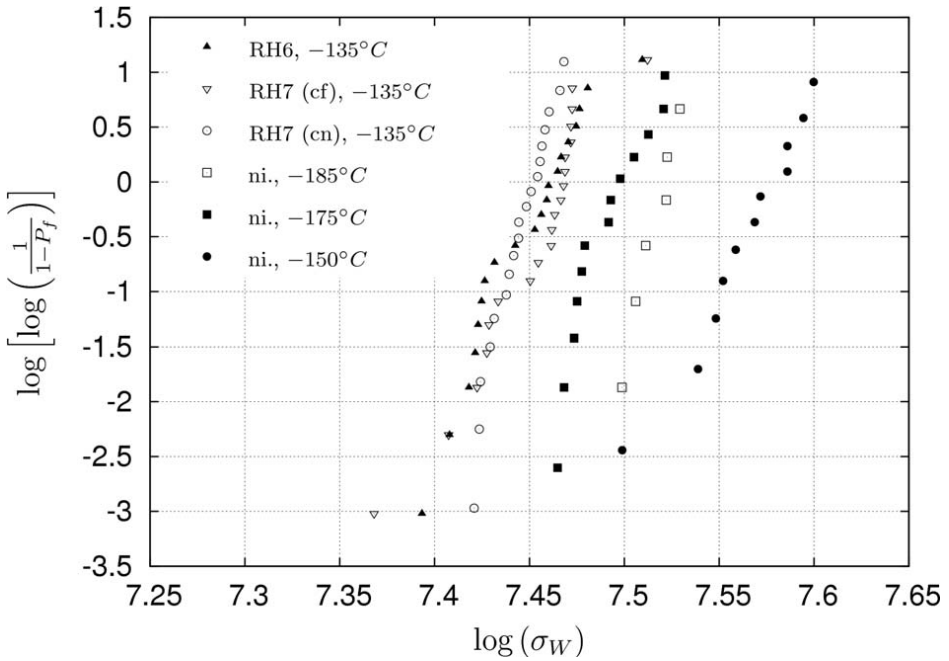


Figure 4: Weibull plots JFL

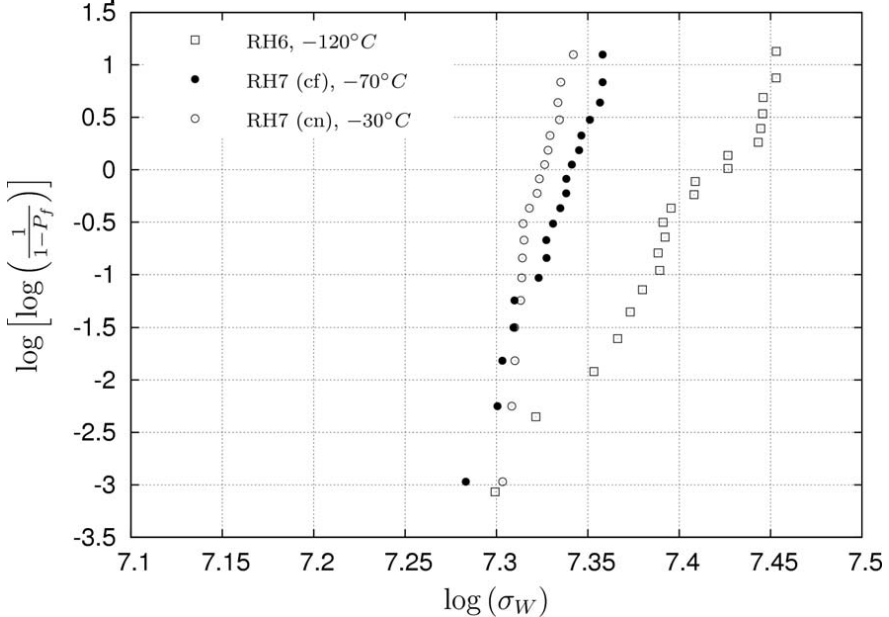


Figure 5: Weibull plots JRQ

**Critical Fracture Toughnesses.** A linear dependency of the Weibull reference stress  $\sigma_u$  on the temperature was assumed for the non-irradiated material JFL. Tests with SENB specimen were simulated for temperatures that defined the brittle and transition region in the SPTs ( $-185^\circ\text{C}$ ,  $-175^\circ\text{C}$ ,  $-150^\circ\text{C}$ ,  $-135^\circ\text{C}$  and  $-105^\circ\text{C}$ ) using FEM and the identified hardening parameters. The

calculated critical fracture toughnesses for 5%, 50% and 95% probability of cleavage fracture are plotted in Figure 6. The values show the expected temperature dependencies. However, the calculated fracture toughnesses are too small for low temperatures, compared with results from Master Curve (MC) tests. Furthermore, a strong mesh sensitivity was observed, since the calculation of Weibull stress requires the actual stress distribution inside the plastified volume of the specimens.

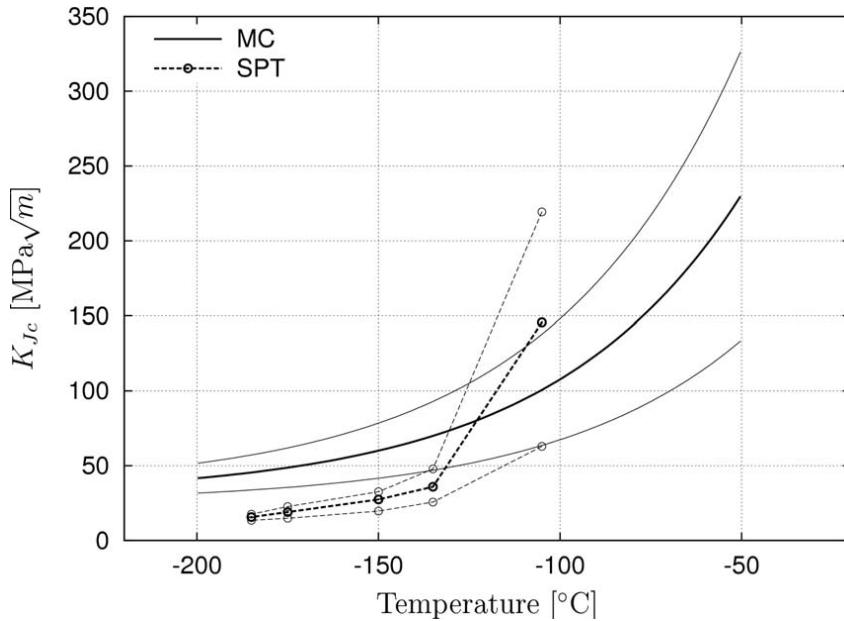


Figure 6: Critical fracture toughness values determined from SPTs.

### Summary

Small punch tests were performed to characterize mechanical properties of two ferritic reactor vessels steels. The required material, both non-irradiated and irradiated, was sampled from remnants of already tested Charpy specimens.

Yield curves were identified from the measured load displacement curves by a global optimization algorithm, in which previously trained neural networks approximate the punch force as a function of the hardening parameters. The identified hardening properties were used to determine Weibull parameters by analyzing the stress distribution inside specimens that failed due to cleavage fracture. For the non-irradiated material JFL, critical fracture toughnesses were calculated with FEM and the identified hardening properties. The stress state inside SENB specimens was analyzed to calculate the probability of cleavage fracture using the determined Weibull parameters.

Additional work needs to be done to investigate possibilities to reduce the mesh sensitivity of the calculated probabilities for cleavage fracture and thus the estimated critical fracture toughnesses.

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