Technical Programme

Investigations on crack resistance characteristics and creep notch sensitivity as a function of ductility of Grade 92

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Contents

1 Motivation/Background

The background for initiating the first technical programme "Long-term behavior of Grade 92" in the first quarter of 2024 was the classification of the martensitic steel grade "Grade 92" (X10CrWMoVNb 9-2, material number 1.4901) as "creep-intolerant" in the current ASME Code Case B31 CASE 183 - 5. Since this classification contradicts the operating experience of European power plant and plant operators with long-term stressed high-temperature components made of Grade 92 steel, a working group was established by vbge energy e.V. to systematically investigate the validity of the classification.

In the first technical programme, the Fraunhofer IWM, MPA Stuttgart, and IfW Darmstadt institutes were commissioned to systematically compile data sets on the damage behavior of Grade 92 (material X10CrWMoVNb9-2, P92 / T92) and further metallographic investigations on broken creep specimens on the one hand, and by extensive analysis of available data from over 350 creep tests on over 30 melts of the Grade 92 material type, most of which originate from European manufacturers, and the associated melt information on the other hand, to answer the question of whether the classification of Grade 92 steel as a "creep-intolerant" material by the ASME Boiler and Pressure Vessels Code (BPVC), Code Case 3048, can also be confirmed or refuted on the basis of the data sets available in Germany. The work was accompanied by theoretical considerations on the creep damage tolerance parameter lambda and on the creep ductility of Grade 92 in general.

The main result of the work is that there is currently no clear evidence that Grade 92 steel with standard heat treatment (VdTÜV sheet 552/2, 2021), chemical composition and grain size, exhibits brittle fracture behavior or notch sensitivity that would justify classification as "creep-intolerant." Although the fracture deformation characteristics of the examined creep specimens decrease with longer service life, the microscopic fracture appearance remains predominantly ductile. However, the statements on notch sensitivity are preliminary, as no results from notch time-dependent tests on Grade 92 in the long-term range are available in Germany to date, while the longest running times known from the literature for notched time-dependent samples made of Grade 92 are in the range of approx. 15,000 hours. Furthermore, the creep damage tolerance parameter λ , referred to as the creep intolerance parameter in ASME Code Case 3048, is the ratio of fracture elongation to Monkman-Grant elongation (accumulated elongation in the secondary creep range) and, although motivated by continuum damage mechanics, is not a suitable parameter for characterizing the ductility of a material. It merely describes the ratio between elongation in the secondary and tertiary creep regions. Although the fracture deformation characteristics of the creep tests on Grade 92 decrease significantly in the long-term range relevant to service, it could not be proven that the test parameters test temperature, test stress, or stress duration systematically influence the parameter λ. Furthermore, the hypothesis that higher sensitivity to pore formation also results in lower creep ductility could not be confirmed by the analyses carried out. It was also shown that individual batches of Grade 92 have a significantly higher sensitivity to pore formation than others, whereby FIB sections through creep pores clearly show that pore formation occurs preferentially at various precipitates (not only at boron nitride). It is also noticeable that individual batches of Grade 92 exhibit significantly better fracture properties (higher values for elongation at fracture and neck-in) than others, although, as mentioned above, no direct correlation between pore density or porosity and creep ductility (elongation at fracture, neck-in) can be identified. The reasons for the different behavior are not yet known. For example, in the investigations carried out on melts with high and low fracture deformation characteristics, no clear correlation could be found between the contents of elements or element groups and fracture deformation characteristics. The examination of the homogeneity of the test materials using element mapping did not reveal any indications of segregation/microsegregation.

The evaluations show that in long-term tests on Grade 92, brittle fracture phenomena can occur with low fracture strain values and simultaneously high creep life consumption up to the 1% or 2% time-strain limits with simultaneously good creep resistance (upper scatter band VdTÜV sheet). It remains unclear whether

- low-deformation Grade 92 melts meet the leak-before-break criterion;
- how ductile and brittle batches can be identified at an early stage, and
- whether brittle melts tend to be more susceptible to notch time sensitivity in the long term than melts with high ductility.

The latter includes the question of the extent to which the creep behavior of batches with low ductility differs from that of batches with high ductility under multi-axial stress. This new technical programme

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will specifically address the above-mentioned topics.

2 Knowledge gain

The planned investigations will significantly reduce the current uncertainties regarding the leak-before-break behavior of operationally relevant material conditions, which could also reduce the safety values specified by the responsible monitoring authority for continued operation of the plant. At the same time, the results will provide sound insights into whether or not the leak-before-break criterion is actually exceeded in long-term use for Grade 92 material, which is classified as creep-intolerant by the ASME. This will be an important criterion in the selection of materials for future HT components that are to be operated at T > 600 °C.

In addition, it will be investigated whether the batch-specific ductility properties in long-term behavior can be determined by slow tensile tests (SSRT tests) on defined material states. For this purpose, a method is to be developed with which the microstructure of untreated material can be specifically adapted by thermo-mechanical pretreatment so that it exhibits the same characteristics as microstructures subjected to long-term creep.

Since no clear microstructural causes for the different deformation characteristics of P92 melts have been found to date, further samples will be examined metallographically for possible segregation/microsegregation using element mapping as part of a new technical programme. A comparison of melts with high and low deformation characteristics in the previous project suggests an influence of the number of M_{23} C_6 carbides. The precipitation structure will be investigated in the planned project for M_{23} C_6 carbides, MX particles, and Laves phase. Fracture surface investigations in the SEM will enable the microscopic fracture appearance to be evaluated and thus classified in the DECS diagram.

The accompanying microstructural investigations of AP2 on thermomechanically stressed samples will enable a statement to be made on the comparability of the test results with those of samples subjected to long-term stress.

3 Services provided by the contracted service providers

3.1 Fraunhofer Institute for Mechanics of Materials IWM – Freiburg

"Experimental investigations into leak-before-break behavior and development of a screening method for identifying brittle batches," in accordance with the attached task description.

AP 1: Experimental investigations into leak-before-break behavior

The aim of the work is to experimentally characterize the crack resistance of different material conditions of Grade 92 as a necessary basis for leak-before-break evaluation of components made of Grade 92 using analytical fracture mechanics models.
To this end, fracture mechanics tests (J-R tests) are to be carried out on materials in different states on standard C(T) samples and special small samples. Accompanying tensile tests (strain rate $1 \cdot 10^{-3}$ 1/s) are required to enable evaluation of the J-R tests. Varying the sample geometry in the J-R tests is considered useful for the following reasons:

- Results from C(T) sample tests can be evaluated according to current standards;
- Small samples enable the experimental characterization of creep-damaged material even if it is not possible to take larger C(T) samples;
- A comparison between C(T) and small sample tests is used to derive and validate the constraint correction function for the J-R curve, which is necessary for later transfer of the results to cracked components.

The following tests are planned in detail:

a) Tensile and J-R tests on standard samples, e.g., C(T)25, at different initial states without pre-damage. The initial states should correspond to two material batches with good and poor long-term properties, respectively, for which the long-term behavior (creep tolerance) is known. This should clarify whether differences in crack behavior in the initial state can provide indications of long-term behavior.

   Scope: 2 tensile tests and 2 J-R tests on standard samples per initial state.

b) Comparative J-R tests on small samples of material in the initial state with poor long-term properties to quantify the influence of constraints.

Scope: 2 J-R tests on small samples.

c) Tensile and J-R tests on material taken from unbroken long-term samples (option 1) or material subjected to operating loads (option 2).

Scope: 6 tensile tests (3 material conditions, 2 specimens each),
6 J-R tests (3 material conditions, 2 samples each with crack extensions of approx. 0.2 mm to 0.5 mm).

It must be taken into account that severe plasticization of the specimen in a J-R test is not permissible. However, this can occur when using small specimens with correspondingly small cracks (e.g., from cylindrical blanks with a diameter of 6 mm) and a material with high crack toughness. For this reason, before determining the test concept, one of the following options should be examined using FE simulations based on the results from AP 1a and AP 1b:

- Option 1: J-R tests on small specimens from unbroken creep specimens – if the crack toughness is low
- Option 2: J-R tests on larger standard samples from material subjected to operating loads – if the fracture toughness is high

d) Fractographic investigations of the tested fracture mechanics samples with regard to the interaction of the cracks with creep pores or with regard to crack propagation, preferably intergranular or transgranular.

The exemplary application of a fracture mechanics model existing at Fraunhofer IWM for leak-before-break evaluation of a cracked component is not the subject of the work in this work package, but is readily possible on the basis of the material properties determined in this work package, provided that the component loads are also known.

AP 2: Screening method for identifying brittle batches

The aim is to develop a screening method for estimating long-term creep behavior based on slow tensile tests (strain rate $1 \cdot 10^{-6}\ s^{-1}$).
To this end, two slow tensile tests will first be carried out on two different initial states for which the long-term behavior is known. Preferably, one batch with good long-term properties and one with poor long-term properties should be used. The tests are intended to clarify whether differences in the deformation behavior in the initial state can already provide indications of the long-term behavior under creep loading.

In a second step, a microstructure typical of long-term creep is to be produced by specifically modifying the initial microstructure through thermo-mechanical loading. To this end, a literature review is to be carried out to determine the state of the art in accelerating the microstructure development occurring under creep loading through thermo-mechanical loading in Grade 92. The thermo-mechanical stress deemed suitable will then be applied to two different initial states for which the long-term behavior is known. The microstructure resulting from the thermo-mechanical preloading will be characterized using comparative metallographic investigations (see work plan of MPA Stuttgart). The specific mechanical properties of the thermo-mechanically preloaded materials will be determined by two slow tensile tests and the results will be compared with those from SSRT tests on the respective initial states.

Finally, two slow tensile tests will be performed on two different materials taken from aged specimens. The results will be compared with those from tests after thermo-mechanical preloading.
The planned investigations therefore aim to verify whether similar results can be achieved in the SSRT test as in long-term stress tests by applying targeted thermo-mechanical preloading to the initial material. If this is the case, a screening method for identifying brittle batches could be derived from the results available at the end of the project based on tests on the initial material.

3.2 MPA Stuttgart

AP 1: Further metallographic investigations

At MPA Stuttgart, microstructural investigations using light, scanning, and transmission electron microscopes are planned. The influence of individual phases such as $M_{23}C_6$ carbides, MX particles, Laves phase, and segregation will be examined in more detail. The data determined in the previous project will be expanded and verified. In addition, the macroscopic and microscopic fracture mechanism of will be documented on selected broken samples.

The investigations will accompany the thermo-mechanical tests in AP2 of the IWM, whereby, for example, it will be examined whether the thermo-mechanical preload leads to a microstructure similar to that of samples subjected to long-term stress.

AP 2: Notched time-dependent tests

Furthermore, notch time tests are to be carried out on selected melts, with a total of 6 notch time tests planned (3 material states, 2 samples each for 1000 h and 3000 h). The following material states are to be considered:

- Material condition 1: Initial condition P92
- Material condition 2: Creep test specimen removed at 1 % elongation
- Material condition 3: Creep test specimen removed at 2 % elongation

For the preloaded material conditions, unbroken, uniaxially loaded creep specimens shall be used, which shall be provided with a notch.

The tests should contribute to the evaluation of the material with regard to creep resistance in the presence of notches, in the initial state and after defined preload. The formation of pores should be investigated on metallographic sections in the notch base in comparison with uniaxially stressed creep tests.

4 General

Both vgbe members and non-members are eligible to participate in the technical programme.

The language of the TP is German/English.

The programme will be carried out if at least 5 companies participate.

The total duration of the TP is 10 months.

5 Participation fee

The costs of the technical programme per programme participant are listed in the table below, based on the number of programme participants:

For vgbe member:

Number of participants	Cost [€]
5	30,400.00
6	25,500.00 €
7	21,900.00 €
8	19,400.00
9	17,200.00 €
10	15,700.00
11	14,200.00 €
12	13,200.00 €
13	12,100.00
14	€11,400.00
15	€10,600.00

* plus statutory value added tax

For NON-vgbe members:

Number of participants	Cost [€
5	€33,400.00
6	€28,000.00
7	€24,100.00
8	€21,300.00
9	€18,900.00
10	€17,300.00
11	€15,600.00
12	€14,500.00
13	€13,300.00
14	€12,500.00
15	€11,700.00

6 Compliance

vgbe energy, vgbe energy's contractual partners in relation to this Technical Programme, and participating companies are committed to fair business practices and reject any form of corruption and bribery. On the basis of this understanding, vgbe energy, the contractual partners of vgbe energy, and the participating companies undertake to strictly comply with their respective internal compliance rules and compliance procedures as well as the statutory anti-corruption regulations. vgbe energy, vgbe energy's contractual partners, the companies involved, and their employees therefore undertake not to offer, promise, or grant any unjustified advantages of any kind in connection with the conclusion and execution of this contract or the contractual relationship arising therefrom, nor to demand, promise, or accept any such advantages. vgbe energy, the contractual partners of vgbe energy, and the companies involved also expect third parties involved in the execution of this contract to behave accordingly and undertake to inform them of their obligation to comply with the law.

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Registration form for participation in the technical programme of vgbe energy e.V.

Technical programme	**"Investigations on crack resistance characteristics and creep notch sensitivity as a function of ductility of Grade 92"**
Company:	
Name	
Position	
Address	
Phone	
Email	
Other Comments	

By signing this document, I declare my binding commitment to participate in the above-mentioned technical programme and thereby agree to contribute financially in accordance with the information provided in chapter 5.

Place, date

Signature

Please send the completed form to: **jens.ganswind@vgbe.energy**

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