# Liquation Cracking and Chromium Depletion in Austenitic Welds of Light Water Reactors

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Keys: Liquation Cracking, Chromium Depletion, EPR, Hot Tensile Test, Weld Simulation


## Abstract

Different types of austenitic stainless CrNi-steels were tested in hot tensile and weld simulation tests including two melts of niobium stabilized austenitic steel, three melts of titanium stabilized austenitic steel and one melt of an unstabilized austenite. The stabilized austenites were tested in conventional versions and in optimized nuclear grade versions. The unstabilized austenite was tested in a conventional version.

The hot tensile tests revealed the conventional Nb-stabilized austenites to have the strongest susceptibility to intergranular liquation cracking followed by the unstabilized material A 304. The titanium stabilized qualities (conventional and optimized ones) exhibited no relevant susceptibility to intergranular liquation cracking. The optimized Nb-stabilized austenite showed no relevant susceptibility to intergranular liquation cracking.

The weld simulation tests revealed with respect to the heat affected zone (HAZ) close to the fusion line the unstabilized austenite A 304 to be most sensitive to intergranular stress corrosion cracking (IGSCC) under Boiling Water Reactor (BWR) conditions. The titanium stabilized austenites (conventional and optimized ones) showed a significantly lower susceptibility to IGSCC. Furthermore, the conventional Nb-stabilized austenites proved to be less sensitive to IGSCC than the Ti-stabilized ones. According to the actual state presented here, the optimized Nb-stabilized austenite shows no susceptibility to IGSCC.


## Introduction

As reported in literature e.g. [1] and compiled in [2], intergranular liquation cracks can be originated in weldments during fabrication by improper procedures. This has been observed mainly on melts of niobium stabilized austenite being fabricated in a conventional manner.

In the beginning of the nuclear technology in the USA in weldments at Nb-stabilized austenitic components this type of cracking was observed. Because of this experience in USA this material was replaced by an unstabilized austenite (A 304) [3]. After 2 to 10 years of service there have been detected intergranular cracks in the heat affected zones (HAZ) of these A 304 weldments. The cracks have been classified by extended research programs as intergranular stress corrosion cracking e.g. [4].

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In contrary to the USA in the German nuclear plants, normally stabilized austenites were used to avoid chromium depletion (sensitization). To prevent the above mentioned liquation cracking the chemical compositions of the stabilized austenites and the welding procedures have been improved.

After a couple of years of service, in 1982 some small intergranular cracks have been detected in heat affected zones of circumferential weldments in titanium stabilized austenitic pipes of a German BWR. A more important number of intergranular cracks has been detected in titanium stabilized weldments by nondestructive testing in 1991 [5]. In niobium stabilized weldments only a small number of intergranular cracks was observed. The discussion on the reasons of the cracking cases was characterized by two different points of view: Crack origination during welding or by corrosion in service.

On that account hot tensile and weld simulation tests were performed in order to classify niobium as well as titanium stabilized and unstabilized austenites with respect to their susceptibility to liquation cracking during welding and to intergranular stress corrosion cracking.

## Materials and test procedures

The chemical compositions of seven tested stainless steel pipe materials and one tested stainless steel forged bar material are listed in table 1. There have been examined two stabilized austenites of optimized production X 6 CrNiNb 18 10 S (A) and X 6 CrNiTi 18 10 S (D), five stabilized austenites of conventional production X 10 CrNiNb 18 9 (B, W), X 10 CrNiTi 18 9 (C, E) and X2 CrNiMoTi 17 12 2 as well as one conventionally produced unstabilized steel X 5 CrNi 18 9 (F).

| Material | | Composition [%] | | | | | | | | | Stabilization | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| MPA Code | Type [1)] | C | Si | Mn | P | S | Cr | Ni | Nb | Ti | Nb/C | Ti/C |
| A (R31) [3)] | X6 CrNiNb 18 10 S | 0,027 | 0,42 | 1,02 | 0,026 | 0,004 | 17,6 | 10,0 | 0,29 | <0,01 | 10,7 | |
| B (R33) | X10 CrNiNb 18 9 | 0,060 | 0,41 | 1,83 | 0,029 | 0,006 | 17,6 | 10,8 | 0,60 | 0,01 | 10,0 | |
| D (R32) [3)] | X6 CrNiTi 18 10 S | 0,024 | 0,17 | 1,84 | 0,011 | 0,011 | 18,1 | 12,0 | 0,01 | 0,35 | | 14,6 |
| E (R22) | X10 CrNiTi 18 9 | 0,055 | 0,36 | 1,70 | 0,028 | 0,012 | 18,0 | 9,1 | 0,02 | 0,35 | | 6,4 |
| C (R24) | X10 CrNiTi 18 9 | 0,038 | 0,21 | 1,97 | 0,029 | 0,008 | 17,3 | 11,2 | 0,02 | 0,18 | | 4,7 |
| F (R18) | X5 CrNi 18 9 [2)] | 0,062 | 0,39 | 1,78 | 0,021 | 0,005 | 18,6 | 9,3 | 0,04 | 0,004 | - | - |
| V [4)] | X2 CrNiMoTi 17 12 2 | 0,018 | 0,57 | 1,61 | 0,028 | 0,015 | 16,9 | 10,9 | 0,01 | 0,111 | | 6,2 |
| W | X10 CrNiNb 18 9 | 0,079 | 0,53 | 1,52 | 0,029 | 0,006 | 17,6 | 10,8 | 0,71 | 0,01 | 9,0 | |

[1)] German Standard [2)] A 304 according to ASTM A 213 and A 312 [3)] optimized version
[4)] Mo 2,02 %, forged bar

*Table 1: Chemical composition of materials studied (product analysis)*

The hot tensile and weld simulation tests were performed at MPA Stuttgart by means of a Gleeble 2000 A-A testing system.

By hot tensile tests the deformation behavior of steels (reduction of area) has been determined for temperatures between 700°C up to temperatures near the liquidus. In the upper range of these temperatures the specimens break intergranularly due to liquefied grain boundaries (liquation cracking).

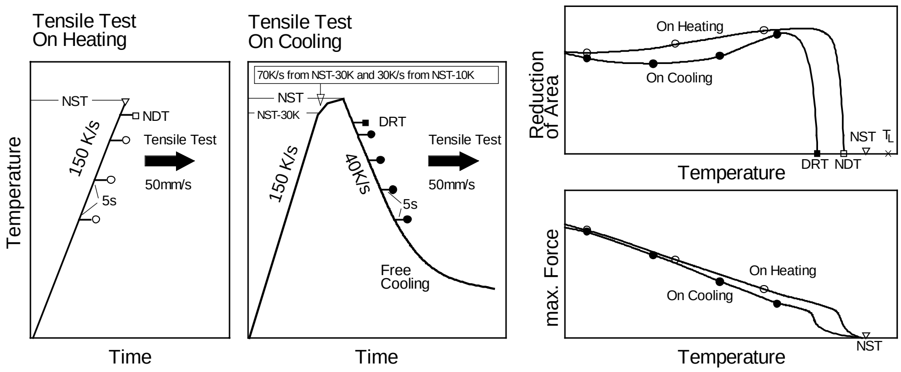


*Figure 1: General procedures of hot tensile test*

**Temperatures**

| | | | |
|---|---|---|---|
| $T_L$ | Liquidus | NDT | Nil Ductility Temperature |
| NST | Nil Strength Temperature | DRT | Ductility Recovery Temperature |

The general procedure of the test is shown in figure 1. When the testing temperature is increased in the On Heating procedure, first the reduction of area increases slightly and then rapidly decreases to zero at the nil ductility temperature (NDT). By further increasing the testing temperature, the nil strength temperature (NST) is reached. At NST the specimens break with a neglectible tension load. Using the On Cooling procedure, figure 1, with declining test temperature at a certain temperature ductility recovers (ductility recovery temperature, DRT).

In hot tensile tests the stainless steels exhibit intergranular fractures with little deformation when the temperature is in the range between NDT up to NST (On Heating) or NST down to DRT (On Cooling). The actual width of the temperature range NST—DRT, where these intergranular liquation cracks are initiated, yields the grade of sensitivity to liquation cracking. To compare sensitivity to liquation cracking of different stainless steels a crack factor with the following formula has been defined as $C_F = (NST—DRT) / NDT \cdot 100$ [%]

Materials with $C_F \geq 4$ % have been classified as “sensitive to liquation cracking“ based on practical experience [6].

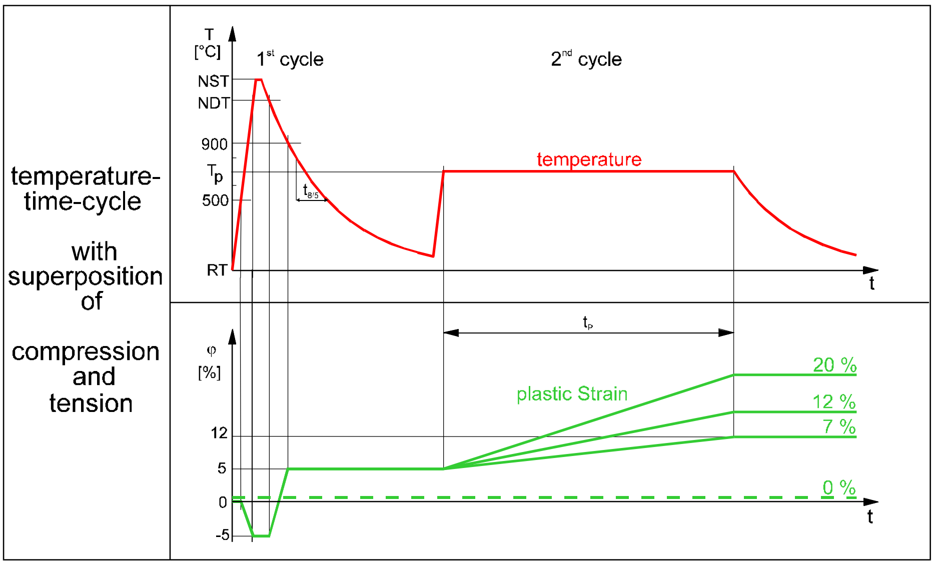


*Figure 2: General procedures of weld simulation tests, temperature and standard strain cycle*

In weld simulation tests (double cycle) specimens are loaded by superimposed temperature and plastic tru strain cycles, figure 2. This test has been developed to produce material micro structures typical for the HAZ of multi pass pipe weldments. The temperature and strain cycles are derived from measurements during multi pass welding by thermo couples and displacement transducers [7, 8, 9]. The 1$^{st}$ cycle of the weld simulation test has a peak temperature corresponding to results of measurements near the fusion line of weldments. Here, dissolution of niobium and titanium carbides takes place and coarse grain is formed. The peak temperature of the 2$^{nd}$ cycle of the weld simulation test is material dependent (range 500—700°C) and calculated [10] by the carbon, niobium and titanium content. For sensitive materials the 2nd cycle leads to chromium depletion of grain boundaries .

The chromium depletion is measured by Electrochemical Potentiokinetic Reactivation (EPR) double loop method and is metallographically documented with an EPR single loop etch followed by metallographic etch to make the grain boundaries visible [11, 12]. The double loop EPR-value $R = I_r / I_a \cdot 100$ [%] shows the degree of susceptibility to IGSCC.

## Results of hot tensile tests

A typical example for the appearance of intergranular fractures at high temperatures is given for a material X10 CrNiTi 18 9 (C) in figure 3.

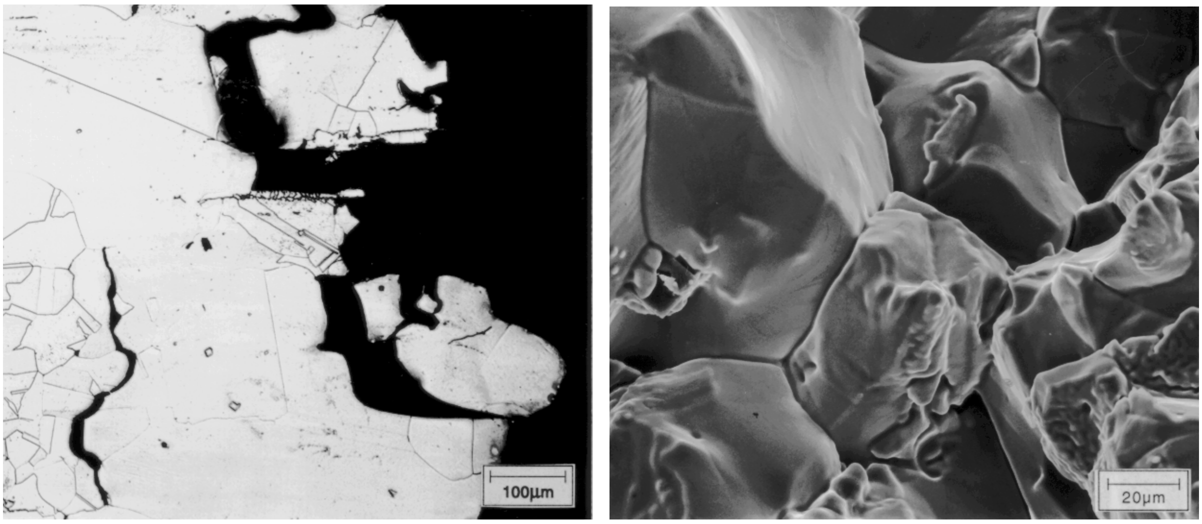


*Figure 3: Metallographic etch and fracture surface of a hot tensile test specimen, material X10 CrNiTi 18 9 (C), test temperature 1371°C (On Cooling)*

Figure 4 shows the results of hot tensile tests on the niobium stabilized austenites A (optimized) and B (conventional). The difference NST—DRT of material A is 53 K ($C_F$ = 3,8 %). Material B has a difference NST—DRT of about 100 K ($C_F$ = 7,4 %). This means that material B is sensitive to liquation cracking in contrast to material A.

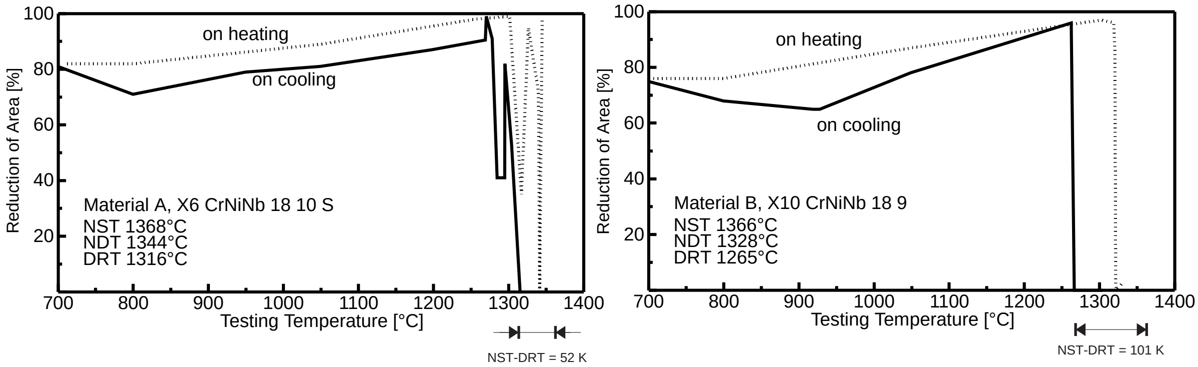


*Figure 4: Reduction of area for different testing temperatures On Heating and On Cooling for the materials X6 CrNiNb 18 10 S (A, optimized) and X10 CrNiNb 18 9 (B, conventional) with corresponding temperature range NST— DRT*

In figure 5 comparable results of the materials G, H, J [6] were added to improve the data base. It becomes obvious that the ferrite content (Ferrite Number FN) is of significant influence on sensitivity to liquation cracking for niobium stabilized austenites. The conventional niobium stabilized austenite (materials B, H, W) is susceptible to liquation cracking. In contrary, the optimized niobium stabilized austenite (materials A, J) is not susceptible to liquation cracking. The material group with the lowest susceptibility to liquation cracking are the titanium stabilized materials. The group as a whole shows no sensitivity to liquation cracking. For these titanium stabilized austenites higher ferrite contents do not contibute to further improvement. The unstabilized materials F and G lie between titanium and niobium stabilized materials right at the 4 % limit of $C_F$.

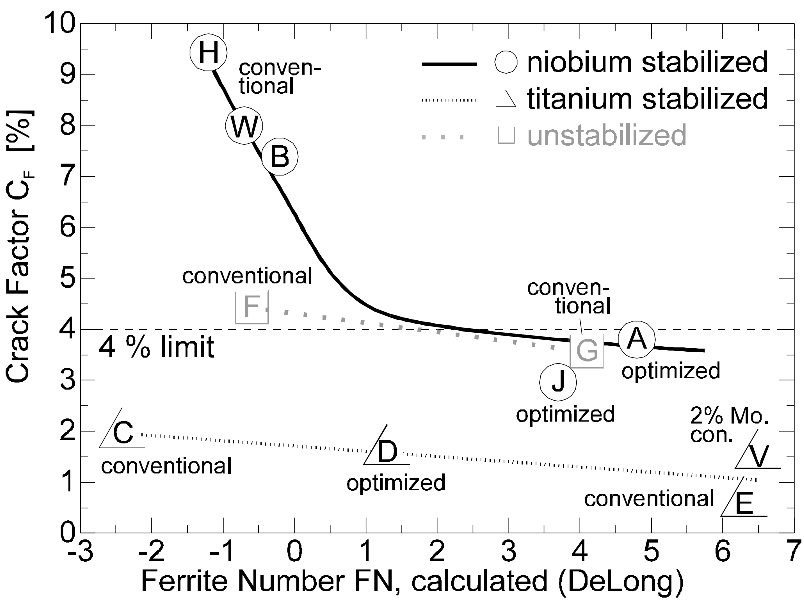


*Figure 5:*
*Influence of the ferrite number on the crack factor $C_F$*
*Materials :*
*A and J - X6 CrNiNb 18 10 S*
*B, H and W -*
*X10 CrNiNb 18 9*
*C and E - X10 CrNiTi 18 9*
*D - X6 CrNiTi 18 10 S*
*V - X6 CrNiMoTi 17 12 2*
*F and G - X5 CrNi 18 9*

**Results of the weld simulation tests**

The weld simulation tests according to figure 2 have been conducted at different plastic true strain levels ($\varphi$ = 0 % $\rightarrow$ 20 %) in the 2nd cycle. When applying about 12 % true strain (intermediate strain level) in the 2nd cycle, the conventional niobium and titanium stabilized austenites B (R=2,0 %) and E (R=2,0 %) show a certain EPR-single loop attack at the grain boundaries whereas the niobium stabilized optimized austenite A (R=0,5 %) does not show

EPR-single loop grain boundary attacks, figure 6. Under the same conditions the unstabilized material F (R=3,0 %) shows the heaviest EPR-single loop attack of all materials.

In figure 7 the main influence of carbon content and of the degree of true strain (φ) on sensitization to IGSCC is shown. The conventional unstabilized material F (A 304) has much higher sensitization levels compared to the conventional, stabilized materials.

The niobium stabilized materials show slightly lower sensitization levels than comparable titanium stabilized ones. The range of EPR-values of all tested austenites is caused by variation of plastic strain in the 2nd part of weld simulation from φ = 0 % to 20 %.

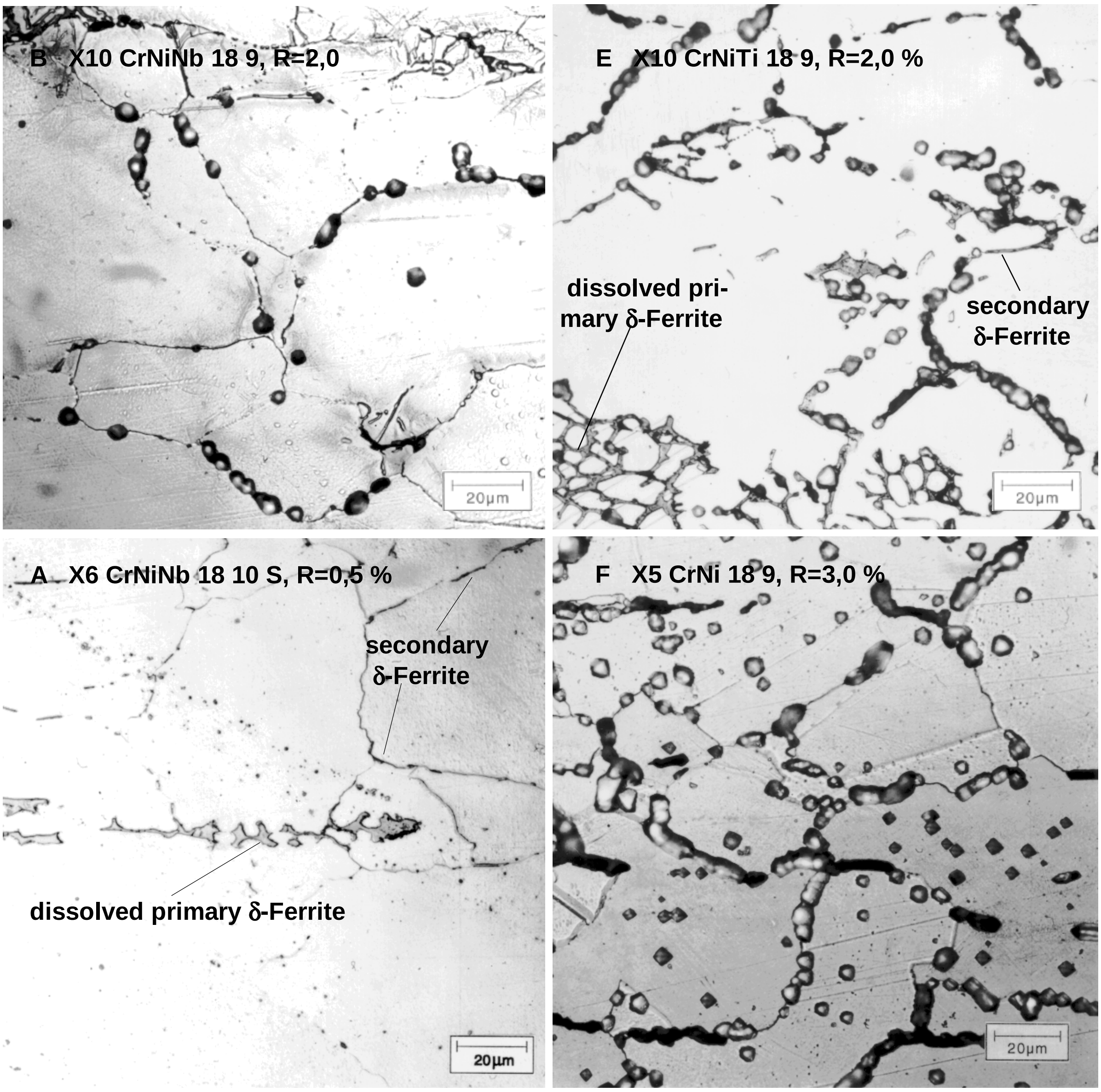


*Figure 6: Weld simulation, intermediate strain, EPR - single loop to show chromium depletion and additional metallographical etch to show the grain boundaries*

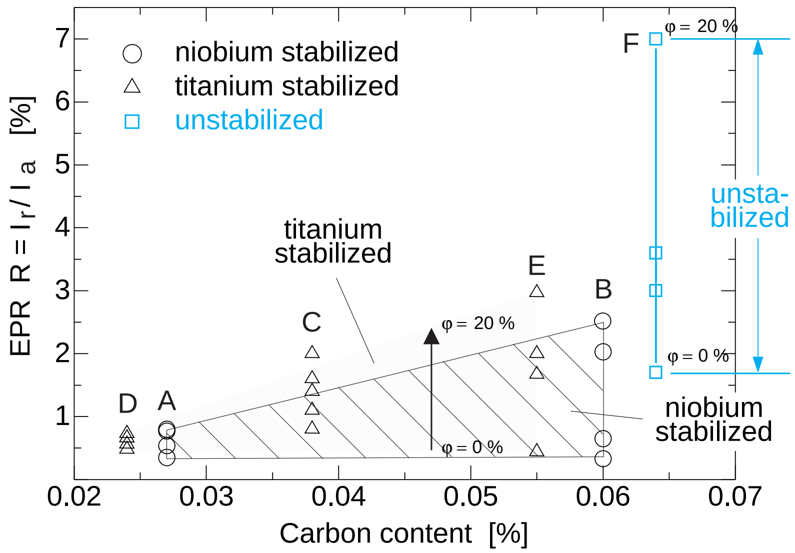


*Figure 7:*
*Influence of carbon content on the sensitization of different groups of austenitic stainless steels, welding simulation (double cycle), $t_{8/5}$ = 45s, plastic strain levels $\varphi$ from 0 % up to 20 % in the 2nd cycle*
*A - 6 CrNiNb 18 10 S*
*B - X10 CrNiNb 18 9*
*D - X6 CrNiTi 18 10 S*
*C and E - X10 CrNiTi 18 9*
*F - X5 CrNi 18 9*

## Conclusions

The HAZ behavior of three different types of austenitic stainless steels with respect to multi pass weldments was characterized by means of hot tensile and weld simulation tests. The actual results are as follows:

Hot tensile tests

- All materials exhibit intergranular fractures (liquation cracks) with practically no reduction of area when tested at high temperatures as occurring in the heat affected zones close to the fusion line.
- From these materials only the conventional niobium stabilized austenite shows susceptibility to liquation cracking with crack factors $C_F$ > 4 %.
- The ferrite content of niobium stabilized austenites proved to be of relevant influence on the sensitivity to liquation cracking. Higher ferrite contents lead to a significantly lower susceptibility to liquation cracking. Titanium stabilized austenites showed no susceptibility to liquation cracking and higher ferrite contents could not contribute to further improvement.
- The optimized niobium stabilized austenite as well as the conventional and optimized titanium stabilized austenite do not show sensitivity to liquation cracking ($C_F$ < 4 %).

Weld simulation tests

- Conventionally fabricated austenites (high carbon content) show generally a certain sensitivity to IGSCC in oxygenated high temperature water:

  Niobium stabilized austenites show a slightly lower sensitivity to IGSCC than comparable titanium stabilized austenites.

  The unstabilized austenite A 304 exhibits a significant sensitivity to IGSCC showing much higher levels of sensitization than conventional stabilized materials.

- The optimized niobium stabilized steel shows no tendency to IGSCC of technical relevance.

As mentioned earlier in stablilized austenitic piping systems of German BWR's a relevant number of cracks in HAZ's of circumferential weldments has been detected by nondestructive examination. The roots of these weldments have not been grinded to remove notches and other root defects. The affected piping systems have been replaced by using the optimized niobium stabilized austenite X 6 CrNiNb 18 10 S [13]. This material with a maximum carbon content of 0,03 % and Nb/C $\geq$ 10 shows neither susceptibility to liquation cracking during welding nor a sensitivity to IGSCC in heat affected zones under BWR conditions.

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