

## The Influence of Flowing Sodium on the Creep Rupture of Notched Welded Specimens of SS AISI 304

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### Abstract

The influence of flowing sodium of 550°C on the creep-rupture behaviour of welded specimens of the austenitic steel AISI 304 (X6 CrNi 18 11) containing a sharp ellipsoidal pre-crack rectangular to the gage length in the welded zone is studied in a sodium loop applying stresses in the range of 140 to 200 MPa.

The life-times of the specimens tested in both media, air and sodium, can be related to the residual stresses in the plane of the pre-crack. The sodium of high purity reduces the time-to-rupture of the welded specimens containing a pre-crack. Strain and fracture are concentrated in the notched weldment. The fracture mode is related to the orientation of the welded layers.

However, the loss of rupture strength (related to the residual stress) is smaller than in preceding in-sodium creep-rupture tests with base material of the same steel, however, from a different batch and in sodium of a lesser degree of purity. A possible mechanism of the influence of sodium and its corrosive action on the crack formation during the creep deformation is discussed.

## 1. Introduction

An influence of flowing liquid sodium on the creep-rupture behaviour of the structural material of the SNR 300, X6 CrNi 18 11 (AISI 304) at 550°C has been observed in earlier tests [1,2]. In sodium tests, the time to rupture is reduced and the fracture is preceded by the formation of numerous intergranular cracks. Though welded specimens behave differently, there is no particular influence of the flowing sodium on the time to rupture. The fracture occurs in the large grain area at both sides of the weldment, and they seem to be the weak points in the specimens [3]. However, the reduction of the life-time of the specimens is smaller in the case, the specimens contain a welding. Defects in the piping of sodium circuits are often located in the surrounding of welded joints. This may be due to the fact that material defects are more often located in the weldings than in the base material. Defected weldings may fail by a faster growing of flaws in the case of superimposure of stress and the action of sodium. To get a more detailed information on the behaviour of defected weldments, a programme was initiated to test the creep-rupture behaviour of welded specimens of the steel X6 CrNi 18 11 in flowing liquid sodium at 550°C. This programme has now reached its final state and results will be presented here.

## 2. Material and fabrication of specimens

The 20 mm thick plate of the austenitic steel X6 CrNi 18 11, the composition of which is shown in the Table I, was cut into stripes. Two of the stripes were prepared for an X-welding by machining one edge in angles of 30°. They were welded together applying the WIG process. The joint was filled with eleven layers. During the filling, the origins for defects were brought in by drilling a fine hole up to 3.5 mm from the center of the plate. At the tip of it we placed a grain of zirconia and closed the cavity before the joint was completely filled up. The particles could be located by X-ray techniques. We cut then rough specimens of 8 mm diameter and developed flaws by oscillating them in the atmosphere. The position and size of these pre-cracks were estimated by eddy current methods. Finally, the creep-rupture specimens of 6 mm diameter and 30 mm gage length were fabricated in a way, that the weldments formed the central part of the gage length. The size of the flaws produced in that way, a typical example of which is shown in Fig. 1, is estimated by measuring the length of the surface crack. The flaws in the 60 specimens of this testing programme were in the order of 10 to 35 % of the cross sections.

## 3. Experimental methods

The creep-rupture tests in air were performed in conventional creep machines. The in-sodium programme could be carried out in a new sodium loop for creep-rupture tests, which has four test sections for the exposure of cylindrical creep specimens under static load. Fig. 2 shows the design of the in-sodium creep machine and the mode of loading and measuring the strain out of the sodium environment. The load cell and the extensometers are located below the heated test section to avoid their heating up. The sodium temperatures at the input and the outlet and also the flow velocity of the sodium were estimated in each of the four sodium lines connected to the testing devices. The flow velocity was maintained at 3 m/s. A by-pass line passed continuously a cold trap operated at  $125 \pm 3^\circ\text{C}$ . Oxygen, carbon and metallic element concentrations were continuously estimated by probes or several times analysed after taking out sodium samples. The impurity concentrations are listed in Table II.

The specimens were degreased and then screwed into the specimen-holder of the test sec-

tions. We closed the test sections under flowing argon and removed the remaining atmospheric gases by evacuation. Afterwards the test sections were heated to the temperature of the main loop (about 350°C), and the sodium passed in after opening the valves. The sodium reduced and washed away all traces of oxides, hydroxides and carbonates still present in the test sections. After a purification period of one day we raised the temperature to the desired value of 550°C and started the test by loading the creep machine after reaching thermal equilibrium. The load was slowly increased to the value producing the desired stress on the specimen. The single test sections could be emptied or refilled during the loop operation.

Strain, load, and temperature were measured at each of the test sections, and all values were collected by the processor. The processor could also be applied to plot the creep curves of individual specimens together with diagrams of the important test parameters. The nominal stress applied was 140, 160, 180, and 200 MPa. Taking into account the remaining cross section in the plane of the pre-crack, the effective stress as residual stress was calculated. After rupture the specimens were taken out of the test sections and examined for their metallurgical changes by several techniques.

#### 4. Test results

After measuring the time to rupture of about 60 specimens we compared the values gained in the in-air and in-sodium tests by selecting groups of specimens in which the applied stress and the size of the pre-cracks were fairly agreeing. Within these groups the residual stress values were of the same order. The creep curves of such groups are used to evaluate differences of the creep-rupture behaviour in both environments. The Fig. 3 compares the creep curves of a typical set of such specimens. As in all cases the specimens exposed to the flowing sodium crack after a shorter time. The creep curves are measured over the whole gage lengths and do not correspond to the creep in the area surrounding the pre-cracks, and the apparent differences of the creep curves cannot be used to estimate secondary creep rates of the strained area of the specimens.

The time-to-rupture of specimens tested in both environments can be related to the residual stresses in the plane of the pre-cracks. This is demonstrated by Fig. 4. The relationships are different for tests in air (eq. 1) and in sodium (eq. 2), indicating the decrease of the life-time due to the action of the liquid metal. The equations fit best the results of all creep-rupture tests of the programme.

$$\log_{10} \sigma_R \text{ (MPa)} = 2.479 - 0.0353 \cdot \log_{10} t_B \text{ (h)} \quad (1)$$

$$\log_{10} \sigma_R \text{ (MPa)} = 2.541 - 0.0649 \cdot \log_{10} t_B \text{ (h)} \quad (2)$$

The time-to-rupture of some specimens without a pre-crack in the welding tested in the sodium loop is only somewhat higher than the values deduced from eq. 2.

The values of strain-to-rupture of the specimens in the both environments do not differ, since only a small part of the gage length contributes to the measured strain. Also the reduction of area seems to be independent on the test medium.

The path of the fracture seems to be influenced by the relative situation of the grains and the welding layers in respect to the pre-crack. Cavities and side cracks can be detected in the grain boundaries along the long dimension of the grains crossing the layers, if the grains are in parallel orientation to the plane of the pre-crack. In the same way the boundary between two welding layers can be the path of the crack development. Therefore, the crack surfaces can be of different topographic appearance. In the case of straight parallel orien-

tation of the grains and the pre-crack plane, the fracture develops in this plane, the creep crack occurs then as the continuation of the pre-crack. On the other hand, pre-crack and fracture are in different planes, if the orientation of grains and layers is not in the plane of the pre-crack. However, all these different fracture modes can be observed in specimens tested in air as well as in sodium.

One of the specimens, removed from the sodium loop before the onset of the tertiary creep, gives a picture of the development of the creep cracks, which cause finally the fracture. In this specimen the pre-crack is opened to a width of 0.3 mm, and from both ends of it some small cracks are growing into the highly deformed weldment.

The fracture mode of the pre-cracked specimens differs completely from that of some reference specimens, which contain intact weldments without any pre-cracks. Though having the fracture life predicted by eq. 2, they develop small cracks along the whole gage length. The fracture is located in the welding zone. However, also in the base material one can see many cracks. The area around the fracture contains a high concentration of such cracks of intergranular character.

The fracture surfaces can be divided into three parts, the initial pre-crack, the surface formed by the growing of the creep cracks during the exposure to the sodium, and the residual fracture in an area of high deformation. The last part is of transgranular mode. In many cases the second part has the same appearance of the fracture surface as the pre-crack. However, also a complete change of the fracture morphology can be observed depending on the orientation of the grains and welding layers as mentioned above.

The metallographic study does not show any typical phenomena of the sodium corrosion of the steel, though under the parameters in the sodium loop the specimens should lose 0.0005 mm/year of their radius due to the solution of material by the liquid metal. Chemical analyses of foils of the same type of steel indicate a slight depletion of carbon and nitrogen out of the specimens. The appearance of their surfaces marks that there has been the sodium-steel chemical exchange, the surface seems to be completely reduced.

## 5. Discussion and conclusions

As indicated by the eq. 1 & 2 sodium reduces the time-to-rupture or the creep-rupture strength of the specimens of welded steel 1.4948 (AISI 304) with pre-cracks. However, the loss of creep-rupture strength observed in these tests is smaller than in the earlier tests with base-material of another batch of the steel [1]. The effect of sodium on the creep-rupture behaviour of the pre-cracked specimens is evident even in the absence of traces of heavy metals dissolved in the liquid sodium. The presence of up to 20 ppm of lead, tin and zinc in the preceding creep-rupture tests in sodium was claimed to be the reason for the loss of strength. Indeed, the effect was smaller in the very pure sodium, in which the notched specimens were tested. Different batches of the steel show some differences of the creep-rupture strength, and also the sodium influence might be different.

Some of the specimens with more than 30 % part of the cross sections pre-cracked and tested with a relatively low load are at the lower border of the scatter-band of the values, which are taken to calculate the eq. 2. From this we can conclude that the effects are somewhat influenced by the size of the remaining cross section. Sodium influences the mechanical behaviour of the specimens by surface reactions. A region of a small cross section has a high relationship of surface to volume. Therefore, an influence of sodium on the time-to-rupture of

such specimens is not surprising. On the other hand, these values are influencing the constants of eq. 2. If we would not include such results into the estimation of the equation, the difference between the eq. 1 and 2 would be much smaller. All the pre-cracks in the specimens in this programme represent a much larger portion of the cross section than the size of the defects that can be safely detected in a component of the fast reactor. We can therefore conclude that the evaluation of our tests leads to conservative relationships.

If we calculate nominal creep rates from the creep curves gained in the tests in air and in sodium, we get different results. It seems that the creep rate in sodium may be somewhat faster than in air. However, we have to consider that the strain is concentrated to a very small area of the gage length. The effects cannot be measured exactly. It seems that a discussion of the creep rates and also the creep strain needs more precise information on the area deformed and its relationship to the whole gage length. The virtual creep rate in the in-sodium tests can be influenced by the development of cracks, which may not be the case in the control tests.

However, there is one effect of sodium on the steel, which can cause a slight loss of strength even in relatively short periods of time. The very pure sodium of our loop causes, as mentioned above, a slight decarburization and denitriding. The intergranular character of the cracks in the tests of base material indicates that the sodium dissolves grain boundary material as precipitated carbides or carbonitrides [2], thus forming germs of cavities between the grains. The cavities formed in that way may be the weak points of the structure, and from here grain boundary cracking starts. Such effects may not be evident, if the sodium does not leach the interstitial elements from the steel. Further investigations have to be done to verify the proposed mechanism of the sodium impact on the high temperature strength of stainless steel. If the action of the liquid metal would take this path, there would be several possibilities to influence the sodium as well as the steel in order to suppress the effect. One can change the precipitates in grain boundaries by changing the thermomechanical treatment of the steel. It is also possible to raise the potentials of carbon and nitrogen in the sodium in order to avoid leaching of the elements and dissolution of grain boundary precipitates.

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#### References

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Table I: Composition of the Stainless Steel X6 CrNi 18 11 (batch 326)

Element	C	Si	Mn	P	S	Cr	Ni	Mo	Ti	N
Conc. (w-%)	.068	.43	1.89	.015	.005	18.92	10.49	.04	.01	.08

Table II: Concentrations of Impurities in the Sodium During the Loop Operation

Element	O	C	Fe	Cr	Ni	Mn	Ca	Zn	Pb	Bi	Sn	Cu
Conc. (w-ppm)	2.5	.15	4	4	.4	.12	3	<1	<.5	<.75	<.9	<.5

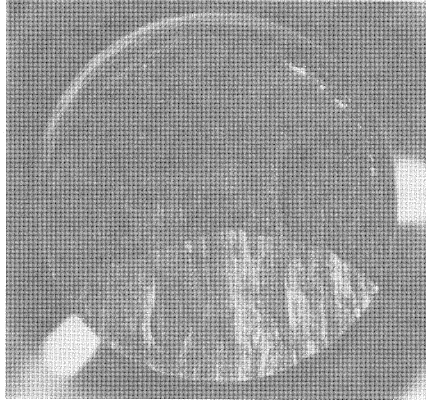


Fig. 1. Crack surface of one of the specimens showing the size of the pre-crack

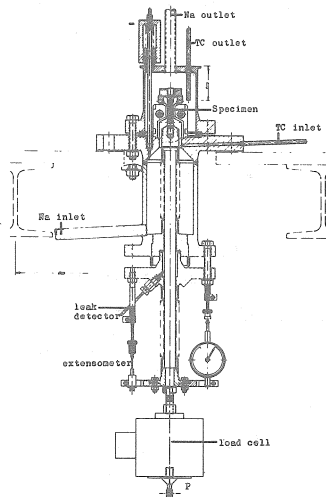


Fig. 2. Creep-rupture test section of the sodium loop CREVONA (sectional view)

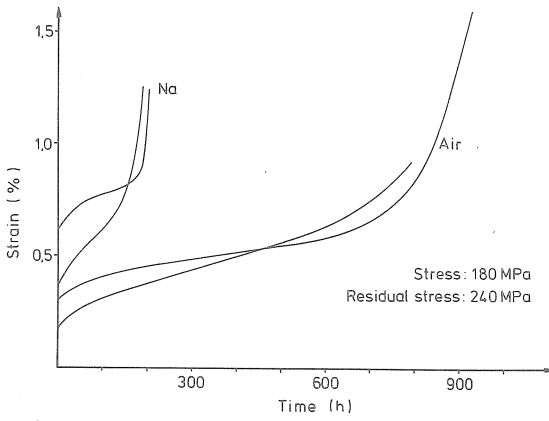


Fig. 3. Comparison of some creep curves or tests in air and in sodium at comparable residual stress (pre-cracked specimens)

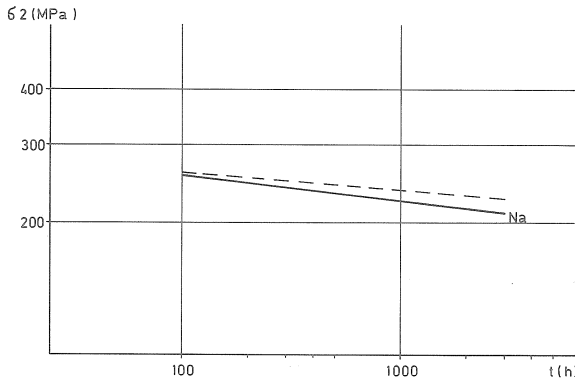


Fig. 4. Time-to-rupture as a function of the residual stress in the pre-cracked specimens

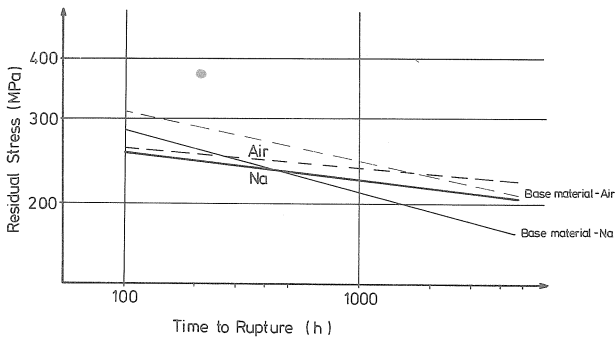


Fig. 5. Comparison of the results with earlier findings (base material)