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# Crack resistance behavior of ODS and standard 9%Cr-containing steels at high temperature

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A B S T R A C T

9%Cr-Oxide Dispersion Strengthened (ODS) steels are under intense research in the EU and other countries to extend the operation temperature range of ferritic alloys towards 600–650 °C. Unfortunately, fracture toughness behavior in this high temperature range is missing. Therefore, the main objective of this work is to evaluate the crack resistance behavior of a 9%Cr ODS steel in the range of 300–650 °C and comparison to the standard nonODS Eurofer-97 steel.

Test results on the ODS steel show a drastic decrease of both initiation toughness and tearing resistance with increasing test temperature. In particular, above 550 °C, the crack resistance curve (*J–R* curve) of ODS Eurofer is extremely low while the standard Eurofer-97 exhibit a much higher crack resistance curve.

## 1. Introduction

9%Cr-Oxide Dispersion Strengthened (ODS) steels are under intense research in the EU and other countries to extend the operation temperature range towards 600–650 °C. An extensive activity aiming to develop an optimum ODS steel with appropriate radiation resistance is in progress worldwide [1–3]. However, their characterization is usually limited to tensile properties, creep strength, Charpy impact and fracture toughness data in the transition regime. Although the ODS steels were designed primarily to operate at high temperatures, typically above 550 °C, fracture resistance describing the material resistance to crack initiation and propagation (tearing) in the ductile regime is often not measured in this temperature range. Also their behavior after irradiation at high temperature (>550 °C) is not well documented. It is only recently that few high irradiation temperature data were published [3].

One of the difficulties when testing at high temperature stems primarily from the instrumentation that is needed to monitor crack extension during the test. However, a number of procedures based solely on the load versus remote displacement record were shown to provide reasonable results and therefore can be used at very high temperatures [4–8]. In particular, we used the energy normalization method [7,8] to estimate the crack growth that is needed to determine the crack resistance curve.

We know that the presence of the fine dispersion also affects the fracture toughness [9] in the low temperature region where fracture is typically brittle. In the ductile regime, there are unfortunately no data available in the literature. Therefore, crack resistance measurements were performed to determine the $J_R$-curve of ODS Eurofer in the temperature range of 300–650 °C.

The objective of this paper is to evaluate the crack resistance behavior of 9%Cr ODS steels in the range of 300–650 °C and to compare the results with those of the standard Eurofer-97 steel. Tensile tests and scanning electron microscopy examination were carried out to complement all these results and support the discussion.

## 2. Materials

The ODS material investigated in this study and denominated “reference EU ODS Eurofer” was produced by Plansee in the form of a hot rolled plate with a thickness of about 6 mm. The chemical composition is given in Table 1.

The characterization of the mechanical properties of the “reference EU ODS Eurofer” (tensile, impact, fracture toughness and creep) in the unirradiated condition was performed within the framework of the European Fusion Development Agreement (EFDA) Work Programme on Structural Materials – High Performance Steels. This report contains the results of the tensile and fracture toughness tests performed at SCK·CEN on specimens extracted from the 6 mm plate (heat HP 1115-6).

The production route started from 200 kg of base alloy produced by Böhler and included: inert gas atomization by Starck (150 kg of powder <150 μm); mechanical alloying of the power with 0.3 wt.% $Y_2O_3$; hot isostatic pressing (HIP) into square bars and finally hot rolling by Plansee, using a cross rolling technique, aimed at achieving homogeneous in-plane properties. Different thermo-mechanical treatments were applied to study their effect on mechanical properties; the plate used in this study was subjected to the following heat treatment: normalization at 1100 °C

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**Table 1**
Chemical composition (wt.%).

| Material | C | Mn | P | S | Si | Ni | Cr | W | V | Ta | Y | Fe |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ODS (0.3% $Y_2O_3$) | 0.07 | 0.41 | 0.011 | 0.03 | 0.11 | 0.05 | 8.92 | 1.11 | 0.19 | 0.08 | 0.19 | bal. |
| Eurofer-97 standard | 0.12 | 0.44 | 0.005 | – | 0.07 | 0.07 | 8.99 | 1.1 | 0.19 | 0.13 | – | bal. |

for 30 min followed by water quenching and tempering at 750 °C followed by air cooling in order to obtain a tempered martensitic structure. Further details on material fabrication can be found in [9–11]. The chemical composition is given in Table 1 where the standard Eurofer-97 is added for comparison.

Transmission electron microscopy (TEM) examination indicated that this material exhibits a quite homogeneous structure with equiaxed grains of 2–8 µm size [12]. The spatial particle distribution of the ODS-particles is relatively homogeneous, with a particle size ranging between about 6 nm to more than 40 nm, the peak being located at around 12 nm. Details of the microstructure can be found in [12].

## 3. Experimental results and discussion

The engineering tensile curves of the ODS Eurofer for various temperatures are shown in Fig. 1. The increase of strength with respect to the standard version is depicted in Fig. 2 which indicates a 200–250 MPa strength increase at all temperatures except above 300 °C after which the difference tends to drastically reduces up to vanishing around 650 °C. The literature data of standard Eurofer-97 were taken from [13].

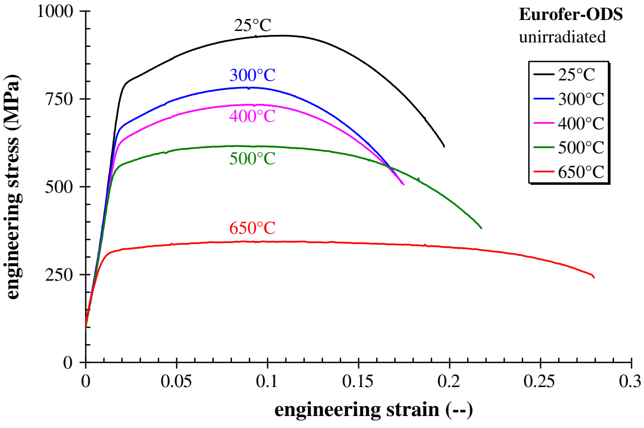

**Fig. 1.** Effect of test temperature on the stress–strain curve of ODS Eurofer.

Because of the limited amount of material, bend specimens were machined from the 6.25 mm-thick plate of ODS Eurofer. Three point bend specimens with a width of 9 mm, a thickness of 6 mm, and a length of 45 mm were machined to optimize the material use. The 2 mm notched (45°, 0.1 mm tip radius) samples were precracked to a crack-to-length ratio close to 0.5 and further 20%-side-grooved to maintain both a uniform crack front and plane strain conditions. The specimens were monotonically-loaded in three-point bending at a constant crosshead speed of 0.2 mm/min, corresponding to a $J$-rate of about 1 kJ/m$^2$ s. The load and total displacement were measured during testing. No instrumentation is used to determine the crack length, the test temperature being too high to attach any device. Instead, a procedure based on the energy normalization is used. This procedure, requiring only the load–displacement curve together with the specimens dimensions and the initial and final crack lengths, was extensively and successfully applied to different materials [7,8]. A typical example of application of such a technique is illustrated on Fig. 3 for standard Eurofer-97 tested at 550 °C. The solid curve is based on one single specimen indicated by an arrow on Fig. 3. As it can be seen, the agreement is quiet reasonable. More details on the procedure can be found in [7,8].

Application of the energy normalization procedure to the four precracked bend specimens of ODS Eurofer tested at 300, 425, 550 and 650 °C leads to the $J_R$ curves shown in Fig. 4. As it can be seen, there is a significant crack resistance degradation when test temperature increases. In particular, at 550 °C and 650 °C, the crack resistance is very low. The low crack resistance of ODS Eurofer at high temperature can also be seen from the crack propagation pattern shown in Fig. 5. At 300 °C, the crack extension pattern exhibits a more zigzag type of crack path than at 650 °C.

Scanning electron microscopy examination of the samples indicate that up to 550 °C the fracture surface is typically dimple, characteristic of ductile fracture but the size of the dimples tends to decrease with increasing temperature (see Fig. 6). At 650 °C, the

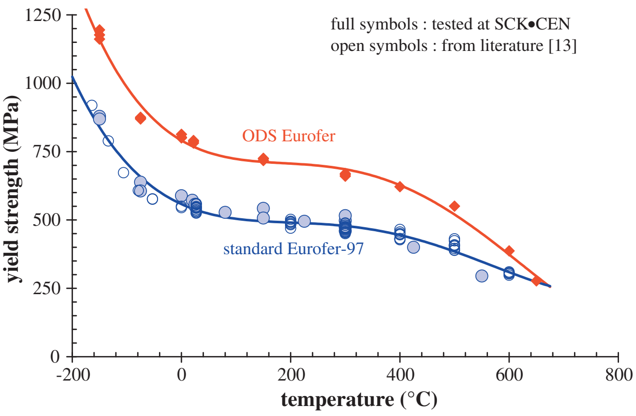

**Fig. 2.** Yield strength–temperature dependence of standard and ODS Eurofer. Literature data are taken from [13].

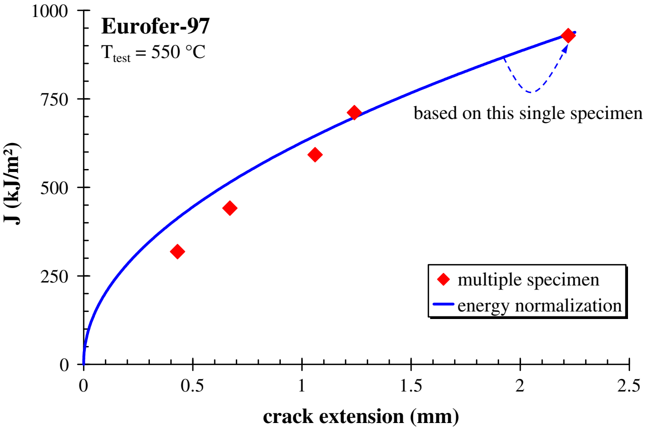

**Fig. 3.** Validation of the $J_R$-curve determination procedure for the standard Eurofer at 550 °C.

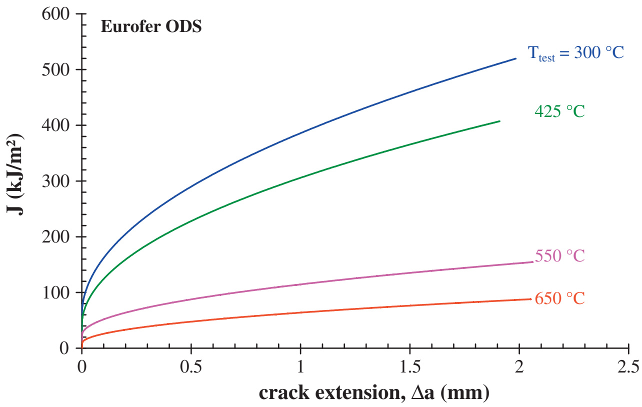

**Fig. 4.** Effect of temperature on the $J_R$-curve of ODS Eurofer.

fracture surface appearance is difficult to characterize, probably intergranular but still ductile (stable crack growth). It is interesting to compare the obtained ODS results to the standard Eurofer steel. As shown in Fig. 2, the strength of the ODS Eurofer is significantly higher than the standard Eurofer. However, from the fracture point of view, the standard Eurofer exhibits a much better toughness than ODS. For ODS steels, the presence of a second phase particle population ($Y_2O_3$ particles) promotes void nucleation, growth and coalescence and therefore facilitating crack propagation. This easy particle–matrix decohesion results from the strain/stress incompatibility between the hard nano-size particles and the relatively soft matrix. A summary of the results is depicted in Table 2 and shown in Fig. 7. This clearly shows that at high temperature, higher strength does not mean high toughness. It is not known how irradiation will affect the crack resistance behavior of ODS steels.

The conventional ODS Eurofer is known to have poor fracture toughness properties [9–11]. In this work, it is shown that its crack resistance at high temperature is also poor. However, there are a number of developments considered to improve the mechanical properties and irradiation resistance of ODS steels. One of the most promising candidates is the so-called nano-structured ferritic steel, where an ultrafine density of nano-clusters is introduced. Very

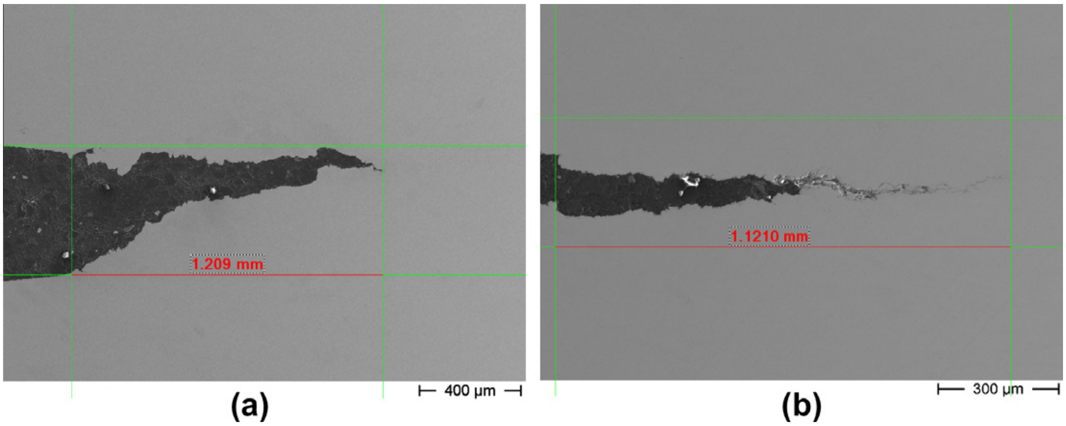

**Fig. 5.** Effect of test temperature on crack extension morphology: 300 °C (a) and 650 °C (b).

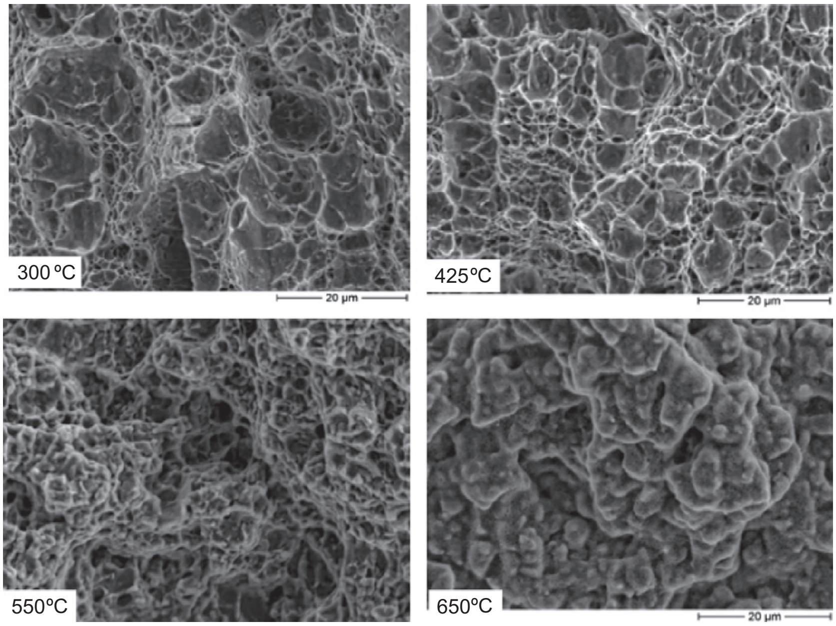

**Fig. 6.** SEM fractography of the fracture surfaces at various temperatures.

**Table 2**
Property comparison between standard and ODS Eurofer.

| Material | Temperature (°C) | $\sigma_y$ (MPa) | $J_{0.2}$ (kJ/m$^2$) | $J_t$ (kJ/m$^2\sqrt{}$mm) |
|---|---|---|---|---|
| Eurofer-97 | 300 | 475 | 342 | 661 |
| Eurofer-97 | 425 | 400 | 394 | 800 |
| Eurofer-97 | 550 | 295 | 341 | 717 |
| Eurofer-97 | 650 | (*180*) | 226 | 483 |
| ODS | 300 | 665 | 205 | 327 |
| ODS | 425 | 600 | 159 | 265 |
| ODS | 550 | 469 | 64 | 92 |
| ODS | 650 | 277 | 33 | 56 |

$\sigma_y$ = yield strength, $J_{0.2}$ is the crack initiation toughness corresponding to the $J$-value at 0.2 mm crack extension and $J_t$ is the tearing resistance derived from the general equation $J = J_i + J_t\sqrt{\Delta a}$ [7,8]. Italic denotes the value obtained as an average between 600 and 700 °C.

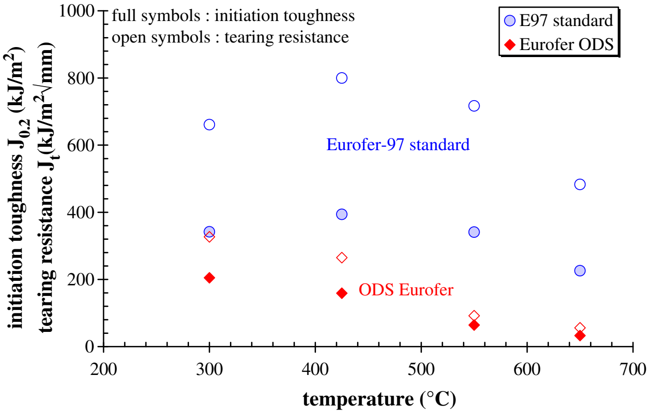

**Fig. 7.** Initiation toughness and tearing resistance versus test temperature of standard and ODS Eurofer steels.

promising results were obtained by McClintock and co-workers [3,14] with a 14%Cr nano-structured ferritic alloy. However, it should me noticed that the irradiation effects reported in [3] are not fully consistent; in particular, the effects of both test and irradiation temperatures are not obvious. More specifically, the slight hardening or even irradiation softening observed on the samples tested at room temperature is not consistent with the significant hardening observed when testing at high temperature. Nevertheless, from the crack resistance point of view, it is very important to perform such tests, before as well as after irradiation at high temperature, to demonstrate their improved performance.

## 4. Conclusion

The 9%Cr ODS Eurofer steel was developed to replace the standard 9%Cr Eurofer to in the temperature range above about 500 °C. Indeed, the presence of the oxide dispersion particles enhances the strength of the steel thereby improving the creep resistance but also the irradiation resistance in particular to helium embrittlement. However, one of the drawbacks of the presence of this oxide dispersion is the low toughness that it induces. But there was no attempt to examine the fracture properties of such materials in the high temperature range where they are supposed to operate. Therefore, the crack resistance behavior of a 9%Cr ODS steel, ODS Eurofer, was investigated in the high temperature range, 300–650 °C. Despite the relatively high strength in this range of temperature with respect to standard (nonODS) Eurofer, the crack resistance is significantly lower than the standard Eurofer. Given the extensive work that is being devoted to the development of ODS steels, including the nano-structured version, it is important to characterize their crack resistance behavior in the high temperature range to effectively demonstrate the good performance of ODS steels before as well as after irradiation.

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