



# Creep-Rupture Properties of Ferritic Cr-ODS Steels in Stagnant Lead at High Temperatures

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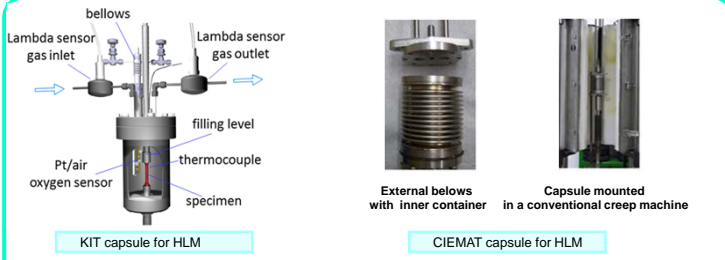
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## Motivation

Intensive study of ferritic and ferritic/martensitic oxide dispersion strengthened (ODS) steels has arisen with increased interest in these materials as candidates for application in nuclear power plants of 4<sup>th</sup> generation and for fusion reactors. High-temperature strength obtained due to randomly dispersed, fine second-phase particles in a metallic matrix by powder metallurgy broadens the application range with respect to temperature. Strengthening mechanisms such as precipitation hardening and solid solution strengthening are less important in the presence of fine dispersoids in comparison to conventional steels. The properties of ODS steels are strongly influenced by the production route, in addition to chemical composition leading to formation of diverse structural compositions or bimodal structures. Ferritic/martensitic 9Cr- as well as ferritic 12Cr- and 14Cr-ODS steels produced by powder metallurgy in form of plates and bars are studied with respect to creep-to-rupture behavior in stagnant, oxygen-saturated lead at 600°C and oxygen-controlled ( $c_o=10^{-6}$  mass.-%) lead at 650°C. Similar experiments were performed in air so as to understand the effect of liquid metal on creep.

## KIT and CIEMAT Creep-Rupture Capsule for HLM. Materials



Determination of the creep-rupture parameters:

$$L_r = L_c + 2 \sum_i [(D/d_i)^{2n} l_i]$$

$$\epsilon_r = \Delta L / L_r \times 100; \quad \epsilon_{r,R} = \Delta L_R^R / L_r \times 100$$

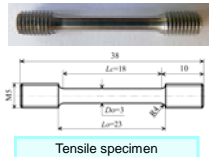


Table 1: Composition (in mass.%) and heat treatment of the extruded 9Cr-ODS plates

Si	Mn	Cr	Mo	V	Fe	Y <sub>2</sub> O <sub>3</sub>
0.67	0.42	8.87	1.66	0.11	87.91	0.32

Thermal heat treatment: hot extrusion at 1150°C, with following forging at 1150°C; finally cold rolling up to 40% reduction and re-crystallized at 1050°C for 1 h; tempering at 750°C for 1 h.

Table 2: Composition (in mass.%) and heat treatment of the extruded 12Cr-ODS plates

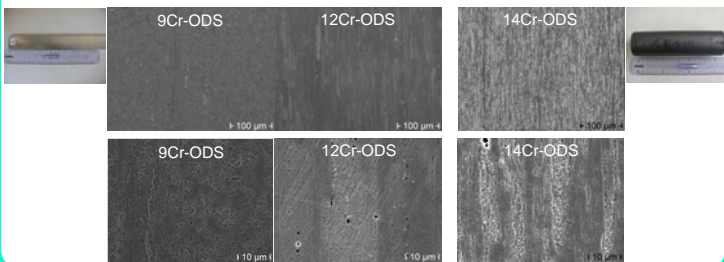
Chemical composition													Calculated	
C	Si	Mn	P	S	Ni	Cr	W	Ti	Y	O	N	Ar	Y <sub>2</sub> O <sub>3</sub>	O
0.02	0.02	<0.01	<0.005	0.002	<0.01	12.2	1.94	0.25	0.17	0.12	0.01	0.005	0.22	0.08

Thermal heat treatment: hot extrusion at 1150°C, with following forging at 1150°C; finally cold rolling up to 40% reduction and re-crystallized at 1150°C for 1 h.

Table 3: Composition (in mass.%) and heat treatment of the cold worked 14Cr-ODS bars

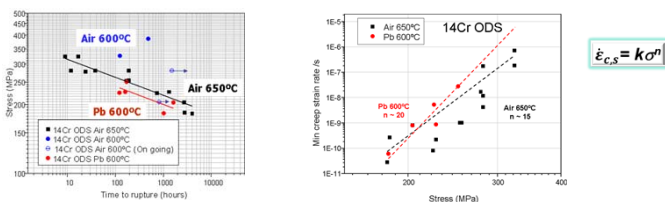
C	Si	Mn	Cr	W	Ti	Y	O
0.41	0.32	0.28	13.46	0.88	0.39	0.22	0.29

Thermal heat treatment: 1050°C for 1.5 h



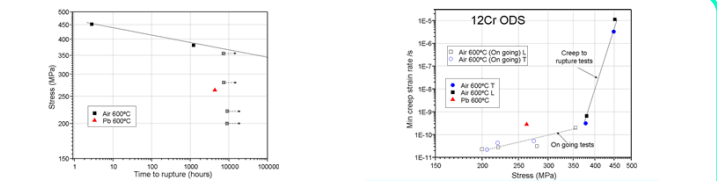
## Creep-Rupture Characteristics

### 9Cr-, 12Cr- and 14Cr-ODSs in oxygen-saturated Pb and air

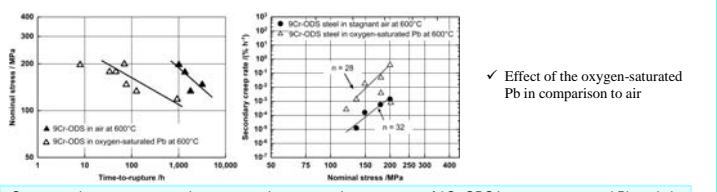


$$\dot{\epsilon}_{c,s} = k \sigma^n$$

- Stress-vs.-time-to-rupture curves of 14Cr-ODS in oxygen-saturated Pb and air at 600-650°C
- Stress-vs.-min creep strain rate curves of 14Cr-ODS in oxygen-saturated Pb and air at 600-650°C
- The Norton exponent  $n$  for the steel tested in air at 180-330 MPa is in accordance to other ODS alloys ( $n=9-15$  [Susila 2011])
- The specimen tested in oxygen-saturated Pb show a higher Norton exponent ( $n=20$ )

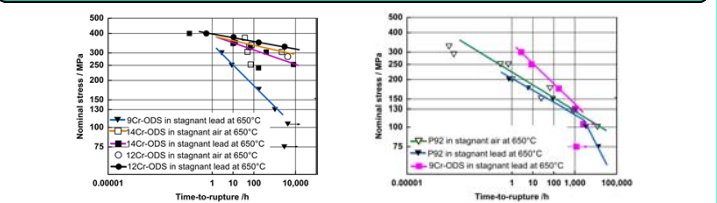


- Stress-vs.-time-to-rupture curves of 12Cr-ODS in oxygen-saturated Pb and air
- Stress-vs.-min creep strain rate curves of 12Cr-ODS in oxygen-saturated Pb and air
- Effect of the oxygen-saturated Pb is observed in comparison to air
- Change of the Norton exponent  $n$  from 7 to 50 by increase of stress suggests a change in creep mechanism

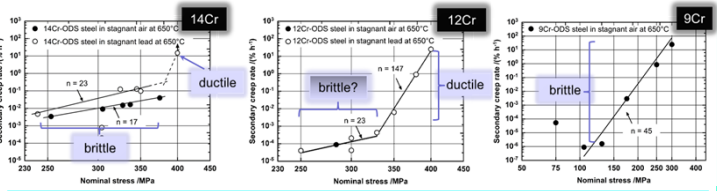


- Stress-vs.-time-to-rupture and stress-vs.-min creep strain rate curves of 9Cr-ODS in oxygen-saturated Pb and air
- Effect of the oxygen-saturated Pb in comparison to air

### 9-14Cr-ODSs in oxygen-controlled Pb (10<sup>-6</sup> mass.%) and air

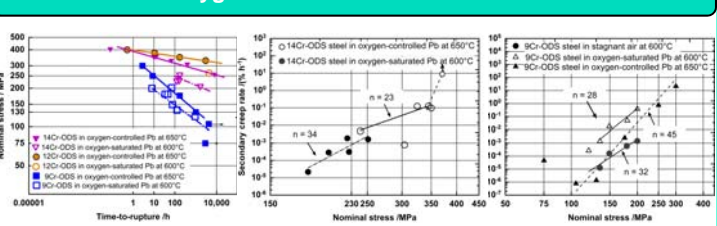


- Strain-vs.-rupture time curves of ODSs in oxygen-controlled Pb and air
- Strain-vs.-rupture time curves of ODSs and conventional P92 in oxygen-controlled Pb and air
- Insignificant difference in strength of 12Cr- and 14Cr-ODS in oxygen-controlled Pb and air at 650°C.
- The lower the stress, the bigger the difference in strength between 12/14Cr-ODS and 9Cr-ODS in Pb (and air).
- The lower the stress, the smaller the difference in strength between 9Cr-ODS and P92 in Pb (and air).



- Stress-vs.-secondary creep rate curves of 9Cr-, 12Cr- and 14Cr-ODSs in oxygen-controlled Pb and air
- 12Cr- & 14Cr-ODSs: Change in fracture mode is affected by deformation rate. Brittle fracture at low load, ductile fracture at high load (ductile-brittle transition around 330 and 350 MPa, respectively).
- 9Cr-ODS: only brittle fracture at 130-300 MPa

### 9-14Cr-ODSs in oxygen-controlled and -saturated Pb at 600-650°C



- 9-14Cr ODSs: high  $c_o$  leads to a stronger decrease of  $t_R$  (even at lower T!) than by lower  $c_o$ . Deformation rate and rate of oxide scale formation (both are higher for oxygen-saturated conditions) could explain this effect.
- Cracks formed on the necking area increase LME risk.
- However, thermal aging effect (microstructural changes) in long-term tests cannot be excluded as a significant degradation factor.

## Summary

- Oxygen-controlled Pb ( $c_o=10^{-6}$  mass.%) and 650°C affects slightly on ODSs with 9-14Cr content in comparison to air.
- Saturation of Pb with oxygen leads to a stronger degradation of the ODSs than in oxygen-controlled Pb even at lower temperature.
- Change in deformation mechanism accompanied by ductile-brittle transition was determined for 12Cr- and 14Cr-ODSs tested in oxygen-controlled Pb and air at 600-650°C, in the stress ranges studied, while 9Cr-ODS features only brittle fracture mode.

## Acknowledgment

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