# The research on oxide dispersion strengthened (ODS) ferritic steel by chemical method

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Iron-based alloys, strengthened with a high number density of nanosized yttrium oxide particles, are typically referred to as oxide dispersion strengthened (ODS) steels, which exhibit better high temperature strength and creep properties than conventional ferritic/martensitic steels while maintaining the attractive properties of this class of alloy such as a low thermal expansion coefficient, high thermal conductivity, and excellent void swelling resistance. ODS ferritic or ferritic/martensitic alloys are being developed for fission applications, in particular as candidate materials for fuel cladding of fast reactor and Gen IV reactors. They are also considered as blanket material for future fusion reactors. However, a challenge in the development of these alloys is maintaining adequate fracture toughness in combination with high strength. According to the literatures (Verhiest et al., 2009; Degueldre et al., 2005; Klueh et al., 2005; Klimenkov et al., 2009; Ukai et al., 2009), ODS ferritic or ferritic/martensitic steels are materials with high temperature strength but inferior impact properties with respect to conventional ferritic or ferritic/martensitic steels.

In our present work, we use a novel chemical processing to generate a homogenous distribution of Y2O3 particles deposited on an argon atomized steel powder, then the composite powder has been consolidated by hot isostatic pressing (HIP), followed by forging and heat treatment. The ODS ferritic steel with nominal composition Fe-12.5Cr-2.5W-0.25Ti-0.2V-0.4Y2O3 (designated 12Cr-ODS) has been fabricated by this manufacturing route, and the microstructure, mechanical properties, hot deformation behavior and (H+/e–) and (He+/e–) irradiation of the ODS steel have been preliminarily investigated.

**Experimental procedures**

The microstructure of the specimens was investigated using transmission electron microscopy (TEM). TEM was performed at 200 kV on a JEOL 2010 equipped with energy dispersive spectrum (EDS) device.

Tensile specimens were machined according to ASTM standard E-8, tensile tests were carried out using an MTS 810 machine equipped with a heating chamber. Round tension test samples of 5 mm diameter and 25 mm gauge length were subjected to tension with a strain rate of $10^{-3}$ $s^{-1}$.

V-notch KLST specimens were machined with the dimensions of $10^{T}$ mm×$10^{W}$ mm×$55^{L}$ mm, 2 mm notch depth, 0.25 mm notch root radius, and 45° notch angle.

Fracture surfaces of the tensile and Charpy impact samples were examined by scanning electron microscopy (SEM) equipped with an energy dispersive spectrometry system.

The hot compression tests were performed using the Gleeble-1500 thermal stimulation machine at constant true strain rates of 0.002, 0.02 and 0.2 $s^{-1}$ and at temperatures of 1070, 1120 and 1150°C.

The effects of (H+/e–) and (He+/e–) dual-beam irradiation on the microstructure damage behavior and oxide particles stable were studied using a combined irradiation device connected by a high energy ion accelerator with a hyperbaric electron microscope.

## Results

***Microstructure.*** Hot rolling results in a heterogeneous microstructure, the typical duplex (martensite plus a-ferrite) microstructure of the 12Cr material is observed. Transmission electron microscopy reveals uniformly distributed grains and a non-uniform dispersion of oxide particles, as shown in Figure P21a. A refinement of the microstructure consisting of equiaxed grains is observed for severely hot forged alloy. In the as-forged ODS material, a fine dispersion of $Y_2O_3$ particles was found with sizes ranging from 10-100 nm. The TEM image in Figure P21b shows nanoparticles that seem to be quite homogeneously distributed in the inner region of the grains, although they were occasionally found on grain boundaries. After forging, the distribution of the oxide particles changes from cluster to dispersed, therefore, forging is an effective way to eliminate the agglomeration and non-uniform distribution of $Y_2O_3$ particles throughout the matrix to obtain dense and uniform alloy, which would contribute to the improvement in the mechanical properties.

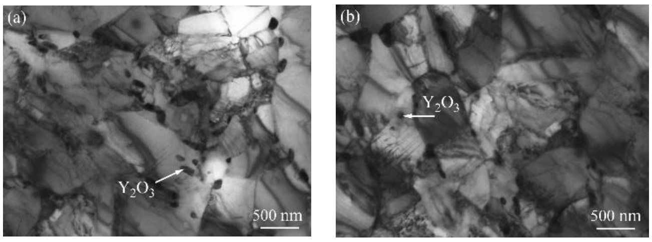


Figure P21: Bright field TEM images showing matrix grains and yttria dispersoids of (a) as-rolled alloy and (b) as-forged alloy.

***Tensile properties and Impact properties***. The results of tensile testing obtained for the 12Cr-ODS alloys in hipped, rolled, and forged conditions are presented in Table P2. Forging or hot rolling significantly improves the mechanical properties of the as hipped ODS ferritic steel. The as rolled ODS ferritic steel has highest yield strength, ultimate tensile strength, hardness, and bending strength. At room temperature the as rolled alloy exhibits ultimate tensile strength and the uniform elongation of 1147 MPa and 7.0%, respectively; The as forged alloy has excellent mechanical properties, at room temperature the ultimate tensile strength and the uniform elongation were 1104 MPa and 10.9%, respectively. At 550°C, The ultimate tensile strength and the yield strength have a slight decrease as the temperature increases to 550°C, and this decrease in strength is accompanied by an increase in uniform elongation. The as-forged 12Cr-ODS steel exhibits very attractive Charpy impact properties with upper shelf energy of 22 J and a low ductile-to-brittle transition temperature of about −15°C.

Table P2: Tensile properties of the 12Cr-ODS alloys tested in different conditions.

| Temperature (°C) | As-hipped alloy | | | As-rolled alloy | | | As-forged alloy | | |
|---|---|---|---|---|---|---|---|---|---|
| | YS (MPa) | UTS (MPa) | UE (%) | YS (MPa) | UTS (MPa) | UE (%) | YS (MPa) | UTS (MPa) | UE (%) |
| RT | 453 | 623 | 2.9 | 763 | 1147 | 7.0 | 738 | 1104 | 10.9 |
| 550 | 406 | 589 | 3.1 | 713 | 1109 | 9.5 | 685 | 1052 | 11.4 |

***Hot deformation behaviour.*** The deformation behavior of the as forged ODS ferritic steel has been investigated by using isothermal hot compression tests. The true stress-true strain curves reveal character of steady state type, which can be represented by a Z parameter in the hyperbolic-sine constitutive equation. The activation energy for hot deformation of the ODS ferritic steel is about 384.487 kJ/mol. The processing maps of the material are obtained according to the dynamic materials model, as shown in Figure P22. The map exhibits two domains: the stability domain in the temperature range of 1070-1135°C and strain rate of 0.002-0.02 $s^{-1}$, and the instability domain in the temperature range of 1075-1120°C and strain rate of 0.02-0.2 $s^{-1}$.

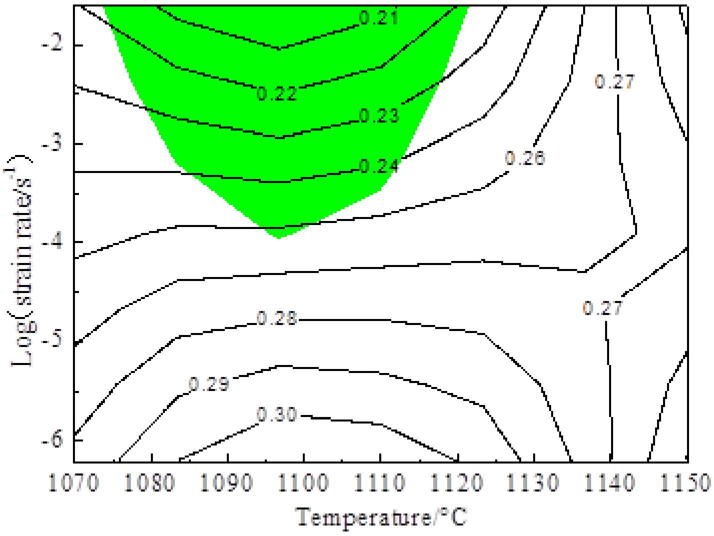


Figure P22: Processing maps for 12Cr-ODS ferritic steel at strain of 0.3, contour numbers represent percent efficiency of dissipation and shaded region corresponds to instability domain.

***($H^+/e^-$) and ($He^+/e^-$) irradiation.*** The as forged ODS ferritic steel shows excellent swelling resistance under ($H^+/e^-$) and ($He^+/e^-$) irradiation at temperature range of 350-550°C, the total void swelling was all less than 0.15% at dose of 15 dpa. The size of the dispersed oxide is stable, no solubility process was observed. Chemical compositions of the oxide particle, the interface, and the matrix have no obvious change before and after irradiation.

## References

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