Comparative Study of Hydrogen Uptake and Diffusion in ODS Steels

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Abstract

- In this work ODS steels and some of their matrix material counterparts, namely, ODS-EUROFER, EUROFER 97, and PM2000 are studied in terms of their interaction with hydrogen at ambient temperature.
- Hydrogen uptake and effective activation energy of its diffusion and trapping are calculated from the thermal desorption spectra obtained for EUROFER 97, ODS-EUROFER, and PM2000 steels.
- It is shown that embedding of ODS nanoparticles lead to significant increase of hydrogen uptake compared to the conventional ferritic steel, such as EUROFER 97.
- It is shown that hydrogen in ODS-EUROFER has the effective diffusion activation energy, which is 0.08 and 0.06 eV larger than that in EUROFER 97 and PM2000, respectively.
- The high-temperature components of the TDS peaks in the studied ODS steels are suggested to reflect hydrogen de-trapping from the oxide nanoparticles.

Materials

	Wt. %										
	C	Si	Mn	Cr	Ni	Mo	Al	W	V	Ti	Co
PM2000	0,017	0,01	0,09	19,3	0,03	0,08	4,8	<0,01	<0,01	0,5	0,15
Eurofer ODS	0,088	0,02	0,4	9,2	0,03	0,02	0,006	1,14	0,18	--	0,12
Eurofer 97	0,103	0,03	0,54	9,0	0,02	0,01	0,019	1,2	0,19	--	0,17

Table 1: Chemical composition of EUROFER 97, ODS and PM2000 steels.

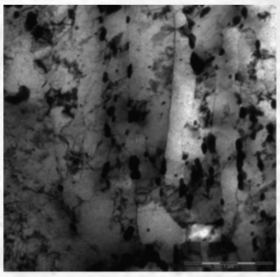
Experimental

Figure 1: TDS apparatus.

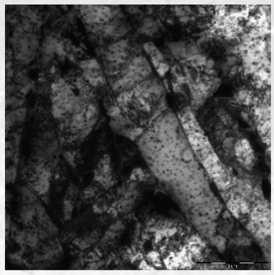
- TDS apparatus was designed and assembled at Laboratory of Engineering Materials of Aalto University:
  - linear heating from RT to 850 °C;
  - heating rates from 1 to 10°C/min;
  - basic pressure in UHV chamber is 10^{-8} mbar.
- Thermostatic enviromental cell for electrochemical hydrogen charging:
  - 0.1N NaOH solution with 20 mg/l of $CS(NH_2)_2$;
  - controlled potential of -1.7 $V_{Hg/Hg2SO4}$;
  - pre-charged with hydrogen for 18 h.
- SEM and TEM techniques.

Results, microstructure

Figure 2: Microstructure of (a) EUROFER 97, (b) ODS-EUROFER, and (c) PM2000 obtained by TEM.



(a)



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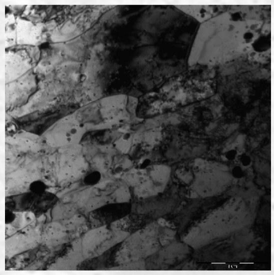

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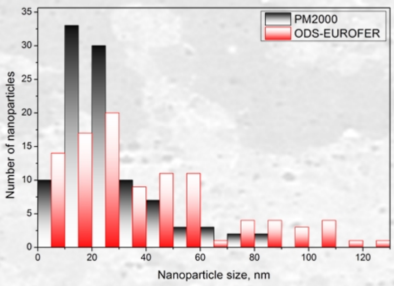
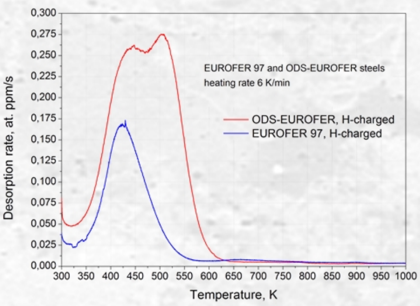


Figure 3: Nanoparticle size distribution for ODS-EUROFER and PM2000.

- Elongated grain structure for all the studied specimens.
- Mean grain size of about 1 μm.
- Mean density of ODS nanoparticles ~ $1\cdot10^{22}$ m^{-3} for EUROFER ODS and $5\cdot10^{21}$ m^{-3} for PM2000.
- The size of the nanoparticles in these materials varied from a few to hundreds nanometers.
- The size distribution analysis of the nanoparticles, having sizes smaller than 100 nm, showed that PM2000 has a smaller mean size of the nanoparticles than ODS-EUROFER, 10 nm vs. 20 nm as shown in Figure 3.
- The EDS line scanning element profile analysis showed that in PM2000 specimens nanoparticles with the sizes of hundred nanometers are aluminium oxide.

Results, TDS



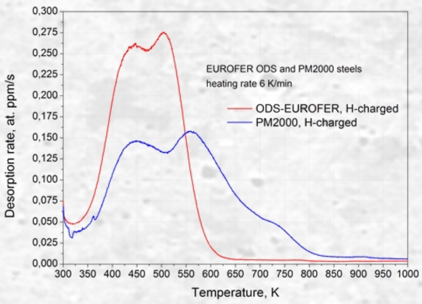


Figure 4: TDS spectra for hydrogen release from EUROFER 97, ODS-EUROFER and PM2000.

The obtained thermal desorption spectra for three heating rates 2, 6 and 10 K/min where used to characterize the binding states of hydrogen in the lattice and activation energies, E_a, of its release from the material.

$$\frac{d\left[\ln\left(\frac{dT/dt}{T_m^2}\right)\right]}{d\left[\frac{1}{T_m}\right]} = -\frac{E_a}{K_b} \qquad (1)$$

Where dT/dt denotes the heating rate, T_m is the temperature of the peak maximum, and k_b is the Boltzmann constant.

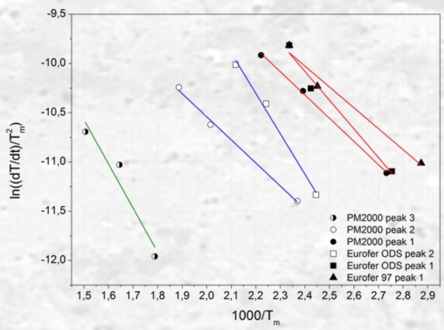


Figure 5: Dependence of $\ln\left(\frac{dT/dt}{T_m^2}\right)$ on 1/ T_m for estimating E_a by Eq.(1).

Activation energy for escape of the hydrogen atom from the lattice trap sites and nanoparticles interfaces can be calculated using liner regression slope of $\ln[(dT/dt)/T_m^2]$ *vs* $1/T_m$ shown in Figure 5.

	Energy, eV		
	Peak 1	Peak 2	Peak 3
PM2000	0,20	0,20	0,38
Eurofer ODS	0,26	0,35	--
Eurofer 97	0,18	--	--

Table 2: Activation energies for the thermal desorption peaks as calculated using Eq.1.

Conclusions

In this work the effect of strengthening of steels by yttrium oxide nanoparticles and their interaction with hydrogen is studied. Hydrogen uptake and effective activation energy of its diffusion and trapping are calculated from the thermal desorption spectra obtained for EUROFER 97, ODS-EUROFER, and PM2000 steels. It is demonstrated that embedding of nanoparticles leads to a significant increase of hydrogen uptake compared to the conventional steel, such as EUROFER 97. It is shown that hydrogen in ODS-EUROFER has the effective diffusion activation energy, which is 0.08 and 0.06 eV larger than that in EUROFER 97 and PM2000, respectively. The high-temperature components of the TDS peaks of the studied ODS steels are suggested to reflect hydrogen de-trapping from the oxide nanoparticles.