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Effect of particle morphology and microstructure on strength, work-hardening and ductility behaviour of ODS-(7–13)Cr steels

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Abstract

The effect of particle morphology and grain refinement to the nanometer scale on strength, work-hardening and tensile ductility of reduced activation ODS-(7–13)Cr steels has been modelled with a dependence on deformation temperature (T = RT–700 °C) and a superimposed irradiation hardening. The Orowan model predictions describe as the upper limit the observed particle strengthening of various ODS-(7–13)Cr-($\leqslant$ 0.5 wt% yttria) steels. An optimum particle size $d_p^* \cong$ 7–22 nm (f_v = 0.004–0.05) and strength, together with a lower limiting ultra-fine grain size $d_{K,c} \geqslant 90$ nm result in maximum uniform ductility increase by grain refinement and dispersion hardening (DIGD). Optimum size d_p^* increases with increasing particle volume fraction f_v and deformation temperature and decreases with irradiation hardening and grain refinement. The region of DIGD is limited to achieve a critical strength σ_L corresponding to a critical particle volume fraction $f_{v,c}$ and grain size $d_{K,c}$, above which uniform strain becomes limited by the strong drop of fracture strain. Grain refinement and irradiation hardening decrease σ_L, $f_{v,c}$ and increase $d_{K,c}$. In accordance with experimental results of ODS-Eurofer, nominal uniform strain increases with increasing f_v by about $\varepsilon_{u,n} = B_e + A_e \ln f_v$, most strongly around 300 °C, but weakly at the 600 °C minimum. The strong ductility increase above 600 °C results from a reduction of dislocation annihilation and structural recovery of strength. At $T <$ 300 °C, grain refinement increases uniform ductility up to $d_{K,c}$ for lower f_v toward a saturation value which increases with increasing ratio of shear modulus to Hall–Petch constant. The enhanced uniform ductility at $T \geqslant$ 300 °C is otherwise strongly decreased by grain refinement, more pronounced at lower f_v and for strengths above σ_L.

1. Introduction

Oxide particle strengthening (ODS) of ferritic–martensitic ODS-(9–13)Cr(Mo)WVTa(Ti) steels increases their uniform ductility, which is contrary to the effect of irradiation hardening. However, this strengthening also decreases the fracture strain and upper shelf energy of Charpy-impact tests due to the enhanced work-hardening [1,2]. Thus, for application of ODS-(7–13)Cr steels as structural material in fusion reactors, they must be optimised by a delicate balance of strengthening achieved by dispersions and irradiation defects with resulting changes of tensile ductility properties and the combined enhancement of dynamic embrittlement.

In this paper, the effects of particle parameters such as volume fraction, size and distribution of particles together with grain refinement to the nanometer scale on strength, work-hardening and tensile ductility of reduced activation ODS-RAFM steels have been modelled as a dependence on deformation temperature (T = RT–700 °C) and a superimposed irradiation hardening. The predictions are compared with experimental results of fine Y_2O_3-oxides (2–40 nm) strengthened ferritic–martensitic ODS-(9–13)CrWVTa(Ti)-(0.1–0.5 wt% yttria) steels. In particular, the optimum particle morphology required to increase the uniform ductility together with the microstructure–property relations are considered for these ODS-RAFM steels. The results are

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particularly useful for optimization and development of ductile martensitic and ferritic ODS-(9–13)Cr steels at combined high strengths.

2. Model of the effect of dispersions on strength, work-hardening and tensile ductility

According to the Orowan dislocation by-pass process [1,2], the strengthening due to non-shearable incoherent particles can be expressed by $\Delta\sigma_p = \beta_{OR}\mu bM(f_v)^{1/2}/d_p$ with the coefficient $\beta_{OR} = 1/(2\pi)\ln\{d_p[\pi/(6f_v)]^{1/2}/b\}$ for screw dislocations. Here, f_v is the volume fraction of uniformly distributed particles of mean size d_p in parallel glide planes, μ the shear modulus, b the Burgers vector and $M \approx 2.5$–3 the Taylor factor for bcc metals dependent on texture. Thus, particle strengthening increases approximately with the square root of particle fraction f_v and more strongly with decreasing mean particle size. The yield strength $\sigma_y = \sigma_{d_K} + \Delta\sigma_p + \Delta\sigma_i$ is given by the linear superposition of solid solution, $\sigma_{0,m}$, and grain boundary strengthening according to the Hall–Petch relation $\sigma_{d_K} = \sigma_{0,m} + k_{HP}/d_K^{1/2}$ with the material constant k_{HP} and the mean grain size d_K, together with dispersion, $\Delta\sigma_p$, and possible irradiation hardening, $\Delta\sigma_i$, due to small defect (clusters, loops) formations. The Hall–Petch relation as found experimentally is generally valid up to Nieh and Wadworth's nano-scale grain size limit for dislocation glide [9], which for Fe is about $\approx$3.4 nm.

Work-hardening by a strain (ε)-induced dislocation population with their density $\rho(\varepsilon)$ can be expressed by $\Delta\sigma_\varepsilon = \alpha\mu bM(\rho)^{1/2}$. The evolution of dislocations can be well described by the modified Kocks-Mecking rate equation of type $\delta\rho/\delta\varepsilon = A - B$ through a balance between dislocation multiplication induced by glide and non-shearable particles/precipitates as well as their annihilation [1,2,10]. Dislocation multiplication according to $A = M\{q\rho^{1/2}/\Lambda_0 + (1-q)/(b\Lambda) + (8/b)f_v/d_p\}$ depends on immobilization of glide dislocations with their mean glide distance Λ and is enhanced by particles due to the generation of geometrically necessary dislocations dependent on particle morphology ratio f_v/d_p. The glide-induced multiplication results by the generated dislocations $\Lambda = \Lambda_0/(b\rho^{1/2}) < d_K$ with part q, where $\Lambda_0 \gtrsim b$ is a material constant and also from grain boundaries as $\Lambda = cd_K$, $c \leqslant 1$ with the remaining portion $(1-q)$. The mean grain size d_K and orientation/distribution coefficient c limit the glide distance. Dislocation annihilation $B = RM\rho/b$ increases with increasing dislocation density ρ and critical distance R below which dislocations of different signs annihilate spontaneously. Annihilation distance $R(T,\dot{\varepsilon}) \geqslant b$ increases with increasing temperature and decreasing strain rate $\dot{\varepsilon}$. For bcc metals at lower $T \leqslant T_0 \cong 0.3T_m$, where glide is mainly controlled by the heavier movable screw dislocations, their glide distance becomes to be bounded by the grain size $\Lambda = cd_K(q=0)$ because they can be easily around sessile dislocations by cross-slips. Above $T \geqslant T_0$ extended edge dislocations mainly control glide, similar to the behaviour of fcc metals with larger grains. By this, dislocations begin to act as strong glide barriers and reduce glide distance according to $\Lambda = \Lambda_0/(b\rho^{1/2}) < d_K$ already within the matrix ($q = 1$), dependently on dislocation density. At very low strains and grain sizes also for fcc $- q = 1$, the work-hardening at first appears according to $q = 0$ with $\Lambda = cd_K =$ const. and toward larger strains according to $q = 1$ by a reduction of the glide distance $\Lambda < d_K$. From this work-hardening model described in detail in [1,10], the results for the strain-induced increase of strength $\Delta\sigma_\varepsilon$ for $q = 0, 1$ are

$$\begin{aligned}
&q=0: && q=1:\\
&\Delta\sigma_\varepsilon = (\Delta\sigma_{\varepsilon,m})^L[1-\exp(-R_m\varepsilon)] && \Delta\sigma_\varepsilon = (\Delta\sigma_{\varepsilon,m})^h[1-\exp(-2R_m\varepsilon)]^{1/2}\\
&(\Delta\sigma_{\varepsilon,m})^L = \alpha\mu M^{3/2}(2R_m\Lambda/b)^{-1/2}[1+8\Lambda f_v/d_p]^{1/2} && (\Delta\sigma_{\varepsilon,m})^h = \sigma_c/R_m\\
& && \sigma_c = [\alpha\mu bM^2/(4\Lambda_0)]\{1+[1+64R_m\Lambda_0^2(bM)^{-1}(f_v/d_p)]^{1/2}\}
\end{aligned}\tag{1}$$

with the corresponding saturation values $(\Delta\sigma_{\varepsilon,m})^L$ at $q = 0$ and $(\Delta\sigma_{\varepsilon,m})^h$ at $q = 1$ for high $\varepsilon \to \infty$ and normalized annihilation coefficient $R_m = RM/b$. The true uniform strain taking into account the plastic stability law $\delta\sigma(\varepsilon)/\delta\varepsilon = \sigma(\varepsilon)$ according Consider [11] is then given by

$$\begin{aligned}
&q=0: && q=1:\\
&\varepsilon_u = -[1/(2R_m)]\ln\{1-\eta_\varepsilon^2\} && \varepsilon_u = -(1/R_m)\ln\{1-[R_m/(1+R_m)][1-\sigma_y/\sigma_c]\}\\
& && \eta_\varepsilon = \{[1+4R_m(1+R_m)y^2]^{1/2}-1\}/[2(1+R_m)y]
\end{aligned}\tag{2}$$

with the strength ratio $y = (\Delta\sigma_{\varepsilon,m})^L/\sigma_y$ and the corresponding uniform ductility maximum at $\sigma_y = 0$ of $\varepsilon^*_{u,0} = [1/(2R_m)]\ln\{1+R_m\}$ for $q = 0$ and

$\varepsilon^*_{u,0} = (1/R_m)\ln\{1+R_m\}$ for $q=1$, dependent only on coefficient R_m, where $(\varepsilon^*_{u,0})^L < (\varepsilon^*_{u,0})^h$. The engineering uniform ductility, which for $q=1$ disappear ($\varepsilon_u = 0$) above a critical yield strength of σ_c is given further by $\varepsilon_{u,n} = \exp(\varepsilon_u) - 1$. The strength ratio $\eta_v = \sigma_{uts}/\sigma_y$ of true ultimate tensile to yield strength describing work-hardening, which correlates with the engineering strength ratio by $\eta_{v,n} = \eta_v/\exp(\varepsilon_u)$, $\eta_{v,n} \leqslant \eta_v$, can be expressed by $\eta_v = 1 + \eta_\varepsilon y$ for $q=0$ and otherwise by $\eta_v = (R_m + \sigma_c/\sigma_y)/(1+R_m)$ for $q=1$. This demonstrates that for both cases $q = 0$,1, the dependence $\eta_v(\varepsilon_u)$ correlates and depends only on R_m [2], as demonstrated also by the relation $\varepsilon_u = -(1/R_m)\ln\{[1/(1+R_m)]\{1+[\eta_v(1+R_m)/R_m - 1]^{-1}\}\}$ valid for $q=1$. The coefficient R_m can be determined directly from the dependences $\eta_v(\varepsilon_u)$, $\varepsilon_u(\sigma_y)$ or the maximum ductility $\varepsilon^*_{u,0}$. Thus, in contrast to $q = 0 (T < T_0)$, for $q = 1 (T \geqslant T_0)$ generally higher uniform ductilities and also η_v – ratios result in lower strengths which, however, reduce and finally disappear ($\varepsilon_u = 0$) above the critical yield strength σ_c (Eq. (1)) up tothe ductile fracture stress σ_{fd}. Because $\varepsilon_u \to 0$ for $\sigma_y \to \infty$ at $q=0$, high strengthened bcc metals ($q=0$) show higher uniform ductilities at comparable strengths than fcc metals, especially at lower temperatures. This ductility can be further enhanced by strong dispersion hardening with high f_v/d_p ratios and also by grain refinement. A saturation of the uniform ductility increase by grain refinement to $d_K \to 0$ is observed for $q=0$, which becomes also valid for $q=1$. It is described by parameter $y^0_{sat} = (\alpha\mu/k_{HP})[bM^3/(2cR_m)]^{1/2}$, independent of whether dispersion, irradiation, or solid solution hardening is responsible. By using y^0_{sat} in Eq. (2), the corresponding achievable saturation ductilities $\varepsilon_{u,sat}$, $\eta_{v,sat}$ can be calculated assuming constant strain rate sensitivity $m = \delta\ln\dot{\varepsilon}/\delta\ln\sigma_y = \text{const} \ll 1$ of yield strength. For low $\varepsilon_{u,sat}$ or μ/k_{HP} values, the simpler upper limit approximations $\varepsilon_{u,sat} \leqslant (\alpha\mu/k_{HP})^2 bM^3/(4c)$ and $\eta_{v,sat} \leqslant 1 + 2\varepsilon_{u,sat}$ are derived. These demonstrate that both parameters $\varepsilon_{u,sat}$, $\eta_{v,sat}$ strongly increase with increasing ratio $(\mu/k_{HP})^2$ but also with increasing $\alpha^2 bM^3$ and become independent of R_m. Toward high μ/k_{HP} values, the increase in saturation ductility $\varepsilon_{u,sat}$ distinctly weakens and finally approaches a maximum value $\varepsilon_{u,sat} \leqslant \varepsilon^*_{u,0} = [1/(2R_m)]\ln(1+R_m)$, which depends only on R_m. For this, the asymtotic solutions $\varepsilon_{u,sat} \leqslant \varepsilon^*_{u,0} - 1/[2y^0_{sat}(1+R_m)^{1/2}]$ and $\eta_{v,sat} \leqslant 1 + y^0_{sat}/(1+R_m)$ become valid. These clearly show, that $\varepsilon_{u,sat}$, similar to $\varepsilon^*_{u,0}$ now additionally reduces with increasing annihilation coefficient R_m by weakening the effectof ratio $(\alpha\mu)/k_{HP}$.

For bcc metals ($q=0$), it results in a uniform ductility increase by grain refinement and dispersion hardening (DIGD) at higher volume fractions $f_v \geqslant f^c_v$ above the critical value $(f^c_v)^{1/2} = 4d_K^{-1/2}\{[\sigma_0 d_K^{1/2} + k_{HP}]^{-1}\beta_{OR}\mu\, bM/c\}$ which is deduced from $\delta\eta_v/\delta\Delta\sigma_p = 0$ at $\Delta\sigma_p = 0$, where $\sigma_0 = \sigma_{0,m} + \Delta\sigma_i$. For low $f_v < f^c_v$ otherwise only the strength-induced uniform ductility decrease is reduced. Therefore, f^c_v strongly decreases by grain refinement and also with increasing irradiation hardening and constant k_{HP} as well as linearly with (μbM). Bcc metals with high shear modulus exhibiting generally higher uniform ductilities but require higher fractions f^c_v corresponding to stronger particle hardening for effective DIGD. Interestingly, for $f_v \geqslant f^c_v$ an optimum particle size d^*_p and strength $\Delta\sigma^*_p$ exists, where maximum ductility $\varepsilon^*_{u,m}$ and $\eta^*_{v,m}$ can be achieved via DIGD. From $\delta\eta_v/\delta\Delta\sigma_p = 0$ for $\Delta\sigma_p > 0$ it yields for the optimum particle size

$$d^*_p = cd_K f_v\{(\sigma_0 + k_{HP}/d_K^{1/2})cd_K f_v^{1/2}/(\beta_{OR}\mu bM) - 1\}^{-1}, \tag{3}$$

which increases with increasing volume fraction f_v but reduces with increasing solid solution, irradiation and grain boundary hardening at larger k_{HP} values. Besides, the achievable ductility increase $\Delta\varepsilon^*_{u,m,n}$ is given by $\Delta\varepsilon^*_{u,m,n} = \{[1-(\eta^*_\varepsilon)^2]/[1-(\eta_\varepsilon)^2]\}^{1/2Rm} - 1$, where coefficients η^*_ε, η_ε relate to conditions of with and without particle dispersions using Eq. (2). Also for fcc – $q=1$ and bcc – $q=1$ at $T \geqslant T_0$, an optimum particle size and hardening are observed for DIGD. The corresponding expressions are, however, more complicated. In Ref. [10] expressions also are given for the fracture strain behaviour with the upper limitation for DIGD by the strength $\sigma_y = \sigma_L$, corresponding to a upper critical volume fraction $f_{v,c}$ and lower grain size $d_{K,c}$, above which uniform ductility increase becomes limited by the strong reduction of fracture strain. It disappears finally at achieve of $\sigma_y = \sigma_{fd}$.

3. Predictions of particle strengthening and uniform ductility increase by grain refinement and dispersion hardening (DIGD) – comparison with experimental results

Fig. 1 shows the observed yield strength increase for various ferritic and ferritic–martensitic ODS-(7–13)Cr-(0.1–0.5 wt% yttria) steels [3–8] as a function of the particle parameter $(f_v)^{1/2}/d_p$, compared with the Orowan predictions using $\alpha = 0.1464$ [2], $M = 2.733$ [2] and $\mu = 82.3$ GPa, $b = 0.25$ nm. The strength increases are obtained from the difference between the measured yield strengths of ODS-steels and base materials with the same composition, produced by the melting process. Volume fraction f_v and mean particle size d_p are measured by TEM examinations or calculated from Y_2O_3-content of $f_v = 1.5618 \times 10^{-2} \times$ (wt% yttria). As demonstrated, the measured strengths $\Delta\sigma_p \leqslant 970$ MPa – $d_p = 3$ nm [7] increases with increasing ratio $(f_v)^{1/2}/d_p$ consistently with the Orowan predictions for uniform distributed particles. However, these Orowan predictions seem to be an upper limit. The deviation of

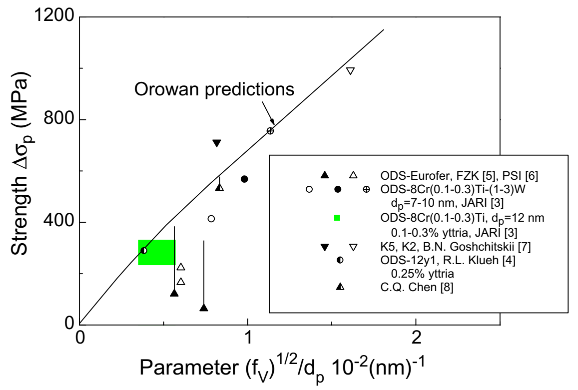

Fig. 1. Comparison of observed particle strengthening of ODS-(7–13)Cr steels [3–8] with Orowan predictions in dependence of $(f_v)^{1/2}/d_p$.

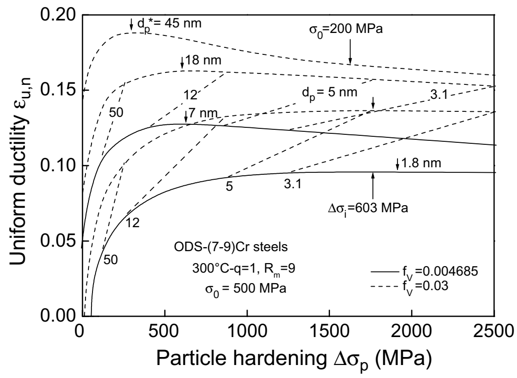

Fig. 2. Predicted effect of particle and irradiation strengthening on uniform ductility increase of ODS-RAFM steels at 300 °C $- q = 1$.

measurements toward lower strengths mainly result from softening due to the ferritic–martensitic duplex structure of ODS-steels compared to fully martensitic base steels. In addition, errors in the determination of f_v and $d_p = 3$–14 nm values and a non-uniform particle distribution, including clustering of small particles also play a role. Such particle clustering reduces Orowan strengthening with maximum amount of $\Delta\sigma_p \propto 1/N^{1/3}$ assuming an initial Gaussian particle distribution, where $N \geqslant 2$ describes the number of particles of same size which form an effectively larger one at constant f_v. For $N = 2$, a decrease of ≈20% results which is similar to that which results from a non-uniform distribution of particles. The measured strength increase of ODS-Eurofer'97 (7Cr,1W,0.2V,0.08Ta) [5] with $\Delta\sigma_p = 243$ MPa for 0.5 wt% Y_2O_3 in the as-received condition is also lower than predictions $\Delta\sigma_p = 432$ MPa using the measured mean size $d_p = 12$ nm of a distribution ranging between 6–40 nm. In that case, the strength deviation mainly results from the weaker matrix strength due to ferrite formation in ODS-Eurofer'97 against the fully martensitic structure of Eurofer'97 by the heat treatment of 950 °C – 30 minV/V + 750 °C – 2 h V/V with vacuum (V) cooling.

As shown already in Ref. [2] an optimum particle size $d_p^* \cong 13$–22 nm and strengthening $\Delta\sigma_p^* = 530$–767 MPa appears for maximum uniform strain increase (DIGD) of ODS-RAFM steels through particle strengthening at $T = \mathrm{RT} - q = 0$ and $f_v = 0.01$–0.05, using $\sigma_0 = 500$ MPa, $R_m = 6$ and $d_K = 5$ μm ($c = 1/5$). With increasing volume fraction f_v, the attainable optimum ductilities $\varepsilon_{u,n}^*$, η_v^* and particle size d_p^* increase in accordance with Eqs. (2) and (3). With increasing irradiation hardening, both values $\varepsilon_{u,n}^*$, η_v^* and d_p^* decrease, whereas $\Delta\sigma_p^*$ strongly increases. As shown in Fig. 2 for ODS-RAFM with 0.3 wt% yttria corresponding to $f_v = 0.00469$ at the higher $T = 300$ °C $- q = 1$ together with $f_v = 0.03$, uniform ductility distinctly increases by particle strengthening also in the case of $q = 1$, being dependent again on morphology parameters f_v, d_p. Compared to $\mathrm{RT} - q = 0$, the DIGD is more pronounced for 300 °C $- q = 1$ due to the combined strong increase of critical strength σ_c. With $d_p^* \cong 7$ nm at $f_v = 0.00469$ and $d_p^* \cong 18$ nm at the higher fraction of $f_v = 0.03$, somewhat lower optimum particle sizes appear with higher strengths $\Delta\sigma_p^* \cong 673$–700 MPa, particularly for lower fractions f_v. With increasing irradiation hardening to $\Delta\sigma_i = 603$ MPa optimum ductility $\varepsilon_{u,n}^*$ and particle size again decrease to $d_p^* \cong 1.8$ nm at $f_v = 0.00469$ and $d_p^* \cong 5$ nm at the higher $f_v = 0.03$, thus, to about similar values as predicted for $\mathrm{RT} - q = 0$. In contrast, $\Delta\sigma_p^*$ strongly increases with a distinct level off of the maximum $\varepsilon_{u,n}^* = 0.088$ toward larger particle sizes $d_p > 1.8$ nm. Thus, for strong irradiated ODS-RAFM steels small particles of $d_p \cong 5$–10 nm seem to provide the best condition regarding DIGD as already observed in Ti-alloyed, Y_2O_3 – particle strengthened ODS-(7–9)Cr steels. The case for a lower matrix strength $\sigma_0 = 200$ MPa at the higher content $f_v = 0.03$ is also shown in Fig. 2, corresponding more to ferritic ODS-steels. It clearly demonstrates, that the ductility maximum becomes distinctly sharper and strongly shifts to lower particle strength ($\Delta\sigma_p^* = 293.5$ MPa) and larger particle size ($d_p^* = 45$ nm) in accordance to Eq. (3). For small fractions $f_v < 0.01$ then, the $\varepsilon_{u,n}^*$ – maximum disappear for unirradiated ODS-steels and particle strengthening now only diminishes the strength-induced uniform ductility decrease. At $\mathrm{RT} - q = 0$ this behaviour appears already at the somewhat lower particle strength (≈80 MPa) corresponding to larger $d_p^* \cong 150$ nm. By the plot $\varepsilon_{u,n}$ vs. ln f_v Fig. 3 shows the predicted influence of the particle volume fraction on uniform ductility of ODS-RAFM again at 300 °C $- q = 1$ for $d_p = 12$ nm. The impact of grain refinement to the ultra-fine size of $d_K = 90$ nm ($c = 1/1.8$) is shown. These are compared

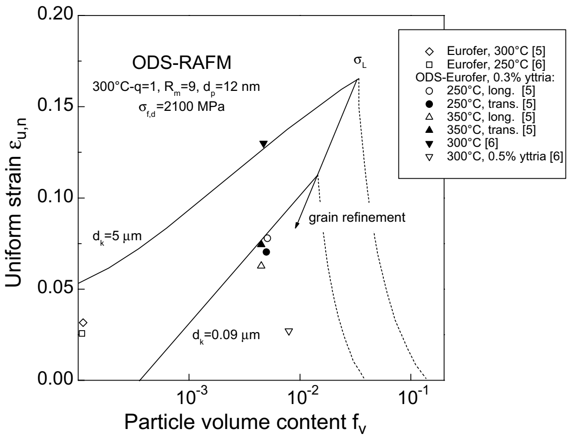

Fig. 3. Predicted dependence $\varepsilon_{u,n}$ vs. $\ln f_v$ for ODS-RAFM at 300 °C $- q = 1$ and $d_p = 12$ nm as function of a grain refinement to $d_K = 90$ nm – comparison with experimental results of ODS-Eurofer'97.

with results obtained on ODS-Eurofer, (0.3–0.5 wt% yttria). Uniform strain for $d_K = 5$ μm continuously increases with increasing f_v up to $\varepsilon_{u,n} = 0.166$ at $\sigma_y = \sigma_L$, corresponding to a larger critical volume fraction $f_{v,c}(d_p) = 0.032$. For higher fractions $f_v \geqslant f_{v,c}$, $\varepsilon_{u,n}$ drastically decreases by the strong reduction of fracture strain ε_f, which limits $\varepsilon_{u,n}$ (i.e. $\varepsilon_f \leqslant \varepsilon_{u,n}$). Grain refinement to $d_K = 90$ nm, taking into account $k_{HP} = 6641$ MPa(nm)$^{1/2}$ gradually reduces $\varepsilon_{u,n}$ due to combined strengthening and additionally decreases critical contents to $f_{v,c} = 0.014$ and $f_{v,f} = 0.042$ at fracture ($\sigma_y = \sigma_{fd}$). It is interesting, that for $5 \times 10^{-4} \leqslant f_v \leqslant f_{v,c}$, the relation $\varepsilon_{u,n}(f_v)$ can be well described by the simple analytic law $\varepsilon_{u,n} = B_e + A_e \ln f_v$ with the dimensionless constants A_e, $B_e > 0$. For $d_K = 5$ μm it yields $A_e = 0.02171$, $B_e = 0.2432$ and somewhat higher constants are observed for the low grain size $d_K = 90$ nm with $A_e = 0.03083$, $B_e = 0.246$. As additionally shown in Fig. 3, the measured $\varepsilon_{u,n}$ values for Eurofer [5,6] and ODS-Eurofer (0.3 wt% Y_2O_3) [5,6] are equal to or below these predictions. For ODS-Eurofer with higher oxide content (0.5 wt% Y_2O_3)[6] the measurements are below predictions which might be due to the onset of local deformation.

Fig. 4(a) shows the predicted grain size dependence of uniform ductility $\varepsilon_{u,n}$ vs. $d_K^{-1/2}$ for ODS- RAFM steels at RT $- q = 0$ and $d_p = 12$ nm as function of f_v (0 to 0.03). The following parameters have been used for calculations: $R_m = 6$, $k_{HP} = 6641$ MPa(nm)$^{1/2}$, $\mu = 83.3$ GPa, $b = 0.25$ nm, $M = 2.72$, $\alpha = 0.1464$ and $\sigma_{fd} = 2100$ MPa. For martensitic structure it is assumed that $c \leqslant 10$ decreases with decreasing d_K and achieves $c = 1$ for $d_K \leqslant 10$ nm. For lower volume fractions $f_v \leqslant 0.05$, most pronounced at $f_v = 0$ (RAFM), uniform ductility con-

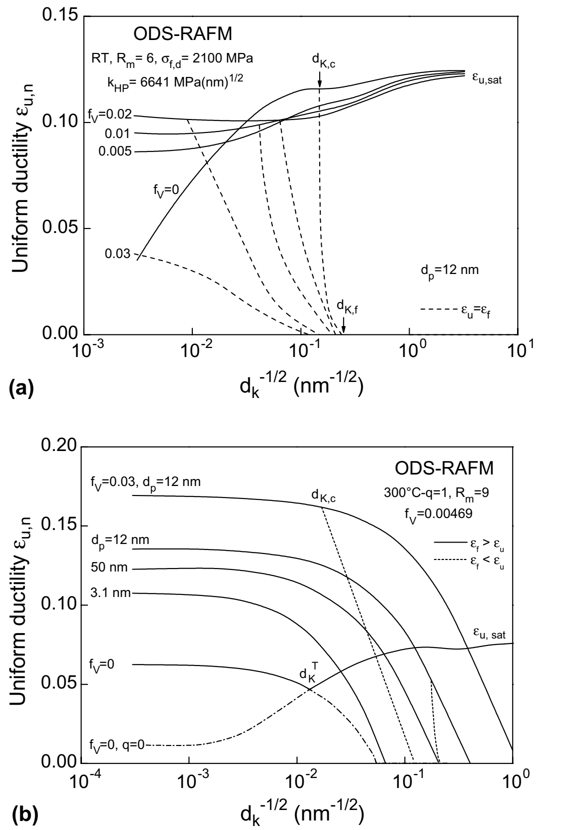

Fig. 4. Predicted dependence $\varepsilon_{u,n}$ vs. $d_K^{-1/2}$ for ODS-RAFM steels (a) at RT $- q = 0$ and $d_p = 12$ nm as function of f_v, (b) at 300 °C $- q = 1$ as function of f_v and d_p.

tinuously increases by grain refinement and tends to saturate at value $\varepsilon_{u,sat} = 0.116$ for nano-scale $d_K \rightarrow 0$. In accordance with predictions, this saturation ductility becomes independent of f_v. At the higher content $f_v = 0.02$, then $\varepsilon_{u,n}$ first weakly reduces due to grain refinement and goes through a broad minimum before it also tends to increase toward $\varepsilon_{u,sat}$. Thus, with increasing fraction f_v, uniform ductility strongly increases up to $f_v \leqslant 0.024$ at larger d_K but decreases weakly at lower d_K. However, when a lower critical grain size $d_{K,c} \geqslant 90$ nm is achieved within the ultra-fine region corresponding to $\sigma_y \geqslant {}_{\sigma}$L, fracture strain drastically drops and limits uniform ductility by $\varepsilon_u = \varepsilon_f$. Finally, it disappears at the nano-scaled grain size $d_{K,f}$, where $\sigma_y = \sigma_{fd}$. With increasing f_v and decreasing d_p, the lower limiting grain size $d_{K,c}$ for DIGD appearance increases more strongly than $d_{K,f}$. At higher volume fractions, as $f_v \geqslant 0.024$ in the case of $d_p = 12$ nm, uniform ductility then becomes limited by $\varepsilon_u = \varepsilon_f$. A strong particle refinement from $d_p = 12$ nm to 3.1 nm restricts the DIGD region to lower values $f_v \leqslant 0.004$. A combined increase of the ductile fracture stress which could be also expected by the grain refinement would reduce both critical grain sizes $d_{K,c}$ and $d_{K,f}$. From the dependence $\varepsilon_{u,n}(\sigma_y)$ obtained for various RAFM steels irradiated at $T_T = T_I = 300$ °C to doses $\leqslant 2.4$ dpa (HFR), a critical yield strength $\sigma_c = 775$ MPa above which $\varepsilon_u = 0$ is estimated. By the measured slope $\delta\varepsilon_{u,n}/\delta\sigma_y = -1/[(1 + R_m)\sigma_c] = S_{\varepsilon,u} = -1.2903 \times 10^{-4}$ MPa^{-1} at strength $\sigma_y = \sigma_c$, the annihilation coefficient $R_m = 9$ is deduced, corresponding to a maximum uniform strain $\varepsilon^*_{u,0,n} = 0.2916$ at $\sigma_y = 0$. Fig. 4(b) shows the predicted grain size dependence $\varepsilon_{u,n}$ vs. $d_K^{-1/2}$ for ODS-RAFM at the higher $T = 300$ °C $- q = 1$ as function of f_v for $d_p = 12$ nm and for different particle sizes d_p at $f_v = 0.00469$ using the derived $R_m = 9$ with the lower modulus $= 76.65$ GPa due to $\mu(T)$. Contrary to RT $- q = 0$, ductility ε_u for $f_v \geqslant 0$ shows a continuous decrease from grain refinement, more strongly at lower d_K and would disappear in case of $f_v = 0$ below $d_{K,c} = 317$ nm up to $d_{K,f} = 15.2$ nm. However, for $f_v = 0$ (RAFM), a transition from $q = 1$ to $\rightarrow q = 0$ occurs at lower grain sizes because glide becomes limited by the grain size $\Lambda = cd_K =$ const., particularly at lower ductilities. Combined with this, uniform ductility then increases again toward to the lower (compared to RT) saturation value $\varepsilon_{u,sat} = 0.0719$ up to the nano-scaled grain size $d_{K,c} = 35.15$ nm for $q = 0$. For ODS-RAFM at higher fractions $f_v \geqslant 0.00469$ and lower $d_p \leqslant 50$ nm, no such work-hardening transition $q = 1 \rightarrow q = 0$ occurs because glide remains always limited by the high dislocation density up to fracture. With increasing f_v (0–0.03) as shown in Fig. 4(b) for $d_p = 12$ nm, uniform strain for $q = 1$ increases also but somewhat more weakly at lower d_K within the ultra-fine region due to the combined strong increase of critical strength σ_c. However, the critical grain size $d_{K,c}$ strongly increases to 3.616 μm at $f_v = 0.03$ for $d_p = 12$ nm. With decreasing particle size $d_p \leqslant 50$ nm as shown for $f_v = 0.00469$ (0.3 wt% yttria), ductility ε_u first increases, more strongly at lower grain sizes within the ultra-fine region. However, $d_{K,c}$ distinctly increases and at last for $d_p = 3.1$ nm, ε_u then strongly drops and becomes always limited by $\varepsilon_u = \varepsilon_f$.

Fig. 5 shows the predicted temperature dependence of uniform strain $\varepsilon_{u,n}$ vs. T for ODS-RAFM steels at $d_p = 12$ nm and RT–700 °C as function of volume fraction $0.03 \geqslant f_v \geqslant 0$ and a grain refinement to $d_K = 90$ nm. The following annihilation constants $R_m = 6/7/21/12$ and critical strengths $\sigma_c = \infty/775/400/362$ MPa have been used at RT/300/600/700 °C as obtained from experimental results on the behaviour of irradiated RAFM taking into account μ(T). As shown, ductility $\epsilon_{u,n}$ of RAFM ($f_v = 0$) for $d_K = 5$ μm first continuously decreases up to the minimum at 600 °C. This reduction occurs by the superimposed effect of a pronounced decrease of shear modulus and both strengths σ_y, σ_c, in combination with the strong increase of parameter $R_m(T)$ due to enhanced dynamic recovery. Above 600

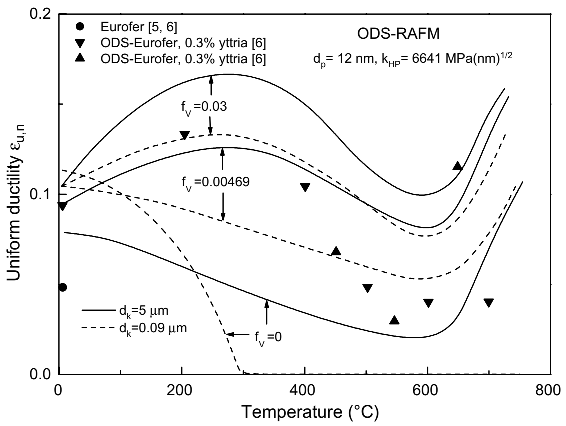

Fig. 5. Predicted dependence $\varepsilon_{u,n}$ vs. temperature of ODS-RAFM at $d_p = 12$ nm as function of f_v and a grain refinement to $d_K = 90$ nm.

°C, uniform ductility then strongly increases mainly by the unexpected reduction of dynamic annihilation coefficient R_m together with a further decrease of strengths σ_c and σ_y. Such reduction of R_m might be caused due to a decrease of the stacking fault energy γ_{st}, appearing already at certain temperatures ≈200 °C below the $A_{c1} \cong 800$ °C point of begin of $\alpha \rightarrow \gamma$ transformation. Dispersion hardening markedly increases $\varepsilon_{u,n}$ with increasing particle volume fraction, most strongly at the mean $T = 300$ °C in accordance with observations on ODS-Eurofer, 0.3 wt% yttria, but somewhat more weakly at the remaining 600 °C- ductility minimum. A minimum value of about $(\varepsilon_{u,n})_{min} = 0.08$ is predicted for $f_v = 0.00469$, which is similar to that observed for ODS-Eurofer,0.3 wt% yttria. At higher $T > 400$ °C, the measured ductilities are somewhat lower than predictions, possible due to onset of local deformation. Thus, the 600 °C uniform strain minimum seems typical for $\alpha \rightarrow \gamma$ transformable steels and can be distinctly increased by dispersion and precipitation strengthening. That increase might be further enhanced by solid solution alloying via a superimposed increase of μ and decrease of γ_{st}, corresponding to a reduction of $R_m(\gamma_{st})$ and the A_{c1} temperature. A strong grain refinement to $d_K = 90$ nm increases uniform strain for RAFM at RT, but would reduce it to $\varepsilon_{u,n} = 0$ for higher $T \geqslant 300$ °C $- q = 1$, because of $\sigma_y > \sigma_c$ due to the strong decrease of $\sigma_c \propto \mu(T)$. However, if the transition $q = 0 \rightarrow q = 1$ occurs at $T \geqslant 300$ °C for RAFM, uniform ductility appears also for the ultra-fine grain size $d_K = 90$ nm. However, the indicated line for $d_K = 90$ nm $- q = 1$ in Fig. 5 remains valid for the strong irradiation ($\Delta\sigma_i = 603$ MPa) strengthened RAFM steels at larger $d_K = 5$ μm and higher irradiation doses. For ODS-RAFM, although the strong strength increase, uniform ductility is only weakly reduced through grain refinement due to high critical strengths σ_c (i.e.$\sigma_y < \sigma_c$). Also, a qualitative similar temperature dependence results as predicted for the larger grain size $d_K = 5$ μm.

4. Summary

(1) Predictions of the Orowan model for uniformly distributed particles by $\Delta\sigma_p \propto (f_v)^{1/2}/d_p$ are the upper limit of observed particle strengthening in ODS-(7–13)Cr-$\leqslant$ 0.5 wt% yttria steels.

(2) Uniform ductility at RT $- q = 0$ and also at higher $T \geqslant 300$ °C $- q = 1$ generally increases by dispersion hardening for strengths below $\sigma_y = \sigma_L$, corresponding to a critical particle volume fraction $f_{v,c}(d_p)$ and grain size $d_{K,c}$. Grain refinement additional increase ε_u (DIGD) particularly at $(T < T_0) - q = 0$.

(3) An optimum particle size $d_p^* \cong 7$–22 nm and strengthening of $\Delta\sigma_p^* \cong 500$–700 MPa appears for DIGD of unirradiated ODS-RAFM steels at RT $- q = 0$ and $T \geqslant 300$ °C $- q = 1$ for $0.004 < f_v \leqslant 0.05$. For both, RT and $T \geqslant 300$ °C, d_p^* increases with increasing f_v but decreases distinctly ($d_p^* \cong$ 1.8–5 nm) by strong irradiation hardening and grain refinement. Otherwise, $\Delta\sigma_p^*$ strongly increases by irradiation hardening and grain refinement and becomes at last limited by $\sigma_y = \sigma_L$ at higher $f_v > f_{v,c}$. Thus, for strong irradiated ODS-RAFM steels at $f_v \lesssim 0.005$ small particles within the range $d_p \cong 5$–10 nm seems the optimum regarding uniform ductility.

(4) Uniform ductility of ODS-RAFM at lower f_v increases by grain refinement at RT $- q = 0$ toward the not attainable saturation value $\varepsilon_{u,sat} = f(\mu, k_{HP}) \neq f_v$, but strongly decreases for higher $T \geqslant 300$ °C $- q = 1$. For RAFM at $T \geqslant 300$ °C $- q = 1$ and lower contents $f_v < 0.004$, the transition $q = 0 \rightarrow q = 1$ occurs at lower grain sizes, after that ε_u again increases up to $\sigma_y = \sigma_L$. Below a critical grain size $d_{K,c} \geqslant 50$ nm within the nano-sized region, $\varepsilon_u = \varepsilon_f$ reduces drastically and finally disappears at $d_{K,f}$.

(5) In accordance with experimental results observed on RAFM and ODS-Eurofer, predicted ductility ε_u first decreases to the minimum at 600 °C by increasing R_m and decreasing strengths σ_y, σ_L. The strong uniform ductility increase above 600 °C occurs then by the distinct reduction of annihilation coefficient R_m. Dispersion hardening generally increases $\varepsilon_u(T)$, more pronounced at $T \approx 300$ °C but also at the 600 °C-minimum. A distinct grain refinement to $d_K = 90$ nm increases generally ε_u at RT $- q = 0$ but reduces it somewhat at higher $T \geqslant 300$ °C, similar to that as predicted for ODS-RAFM steels.

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