Correlation between tensile ductility and impact properties of oxide dispersion-hardened ODS-Ta-W alloys

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Abstract

The effects of dispersion strengthening and grain refinement to the nano-crystalline level (d_k>3 nm) on the tensile ductility and Charpy-impact behaviour have been examined for ODS-Ta≤40at% W and ODS-W alloys by deformation/fracture models. Grain refinement strongly increases the ductile-to-brittle transition temperature DBTT and reduction of upper shelf energy U=∆USE/USE_0 at constant fracture stresses and also reduces uniform ductility. Dispersion hardening increases further DBTT and energy shift U but additionally increases uniform ductility more pronounced at a nanoscale optimum particle size of d_p*≤≤5-40 nm. That reduces with increasing grain refinement, W alloying and decreasing particle volume fraction. A grain refinement-induced increase of fracture stresses as observed in W up to the size d_k≥ 500 nm, however, strongly reduces DBTT and weaker also energy shift U within the ultra-fine(uf) grain region. Minima in DBTT and shift U result at optimum uf grain sizes d_k≥50 nm already at a weak fracture stress increase, which increase with increasing W alloying and dispersion hardening. The deduced optimum W content for ductile and high strengthened ODS-Ta-W alloys are between 20-≤40at% W dependent on dispersion strengthening, grain refinement and induced fracture stress increase.

1. Introduction

Oxide dispersion-strengthened (ODS)-Ta-W alloys due to their high ductility, strength and excellent impact properties are generally proposed as low (<20°C) and high-temperature structural materials for application up to 1300°C. These, besides the more brittle W alloys are especially discussed for application as structural material of the divertor of future fusion reactors to control heat and particles (hydrogen isotopes, helium and impurities) from plasma at operational temperatures of ≤1200-1300°C. High thermal shock, strength and conductivity properties are required for that application together with a reduced hydrogen and helium embrittlement. For comparison, the use of the high strengthened ferritic-martensitic ODS-(7-13)CrWVTa steels is restricted below < 750°C. For optimisation of this ductile, high-strengthened ODS-Ta-W and ODS-W alloys, the superimposed effects of dispersion strengthening and grain refinement to the nano-crystalline (nc) level (d_k>3 nm) on the tensile ductility and Charpy-impact behaviour have been examined by micro-mechanical deformation and ductile/dynamic fracture models [1]. Especially, the effects of dispersion parameters as mean size and volume fraction upon the grain size dependence of the ductile-to-brittle transition temperature DBTT and ductile upper shelf impact energy USE have been investigated taken into account strengthening by ≤40at.% W solid solution alloying and irradiation-induced defect formation. Additionally, also the influence of a combined increase of ductile and dynamic fracture stresses due to grain refinement has been considered which might result from a transition of deformation process from dislocation glide to diffusion-induced grain-boundary sliding. The data used for predictions have been observed by analysing the reported work-hardening behaviour of large grained Ta-(0-20)W alloys [2] and the ultra-fine (uf) grained pure Ta [3] produced by the equal channel angular pressing (ECAP). The predictions are particularly compared with the behaviour for ODS-(7-13)CrWVTa steels and reported results upon the grain refinement effects of the DBTT for pure W [4].
2. Model for the influence of dispersion hardening and grain refinement on tensile ductility and impact behaviour of ODS-Ta-W alloys

The total yield strength of ODS metals is given by the linear superposition

$$\sigma_y = \sigma_{dk} + \Delta \sigma_p + \Delta \sigma_i$$  \hspace{1cm} (1a)

from various contributions like grain boundaries, $\sigma_{dk}$, incoherent dispersions or non-shearable precipitates, $\Delta \sigma_p$, and additionally from small defects $\Delta \sigma_i$ as atomic clusters (<1 nm), dislocation loops and helium bubbles (>1 nm) formed in the case of neutron irradiation within the matrix. Grain boundary strengthening is described by the well-known Hall-Petch relation

$$\sigma_{dk} = \sigma_{o,m} + k_{HP} / d_K^{1/2}$$  \hspace{1cm} (1b)

dependent on the material constant $k_{HP}$ and the mean grain size $d_K$ together with the solid solution contribution $\sigma_{o,m}$ depending on alloying as the W content in Ta-W alloys. It is generally valid for metals up to nc grain sizes of $d_K \approx 3-20 \text{ nm}$ [5] as about $\approx 3.4 \text{ nm}$ for iron. Strengthening due to incoherent particles or larger coherent precipitates above their shearable limit can be expressed according to the Orowan dislocation by-pass process [1] by

$$\Delta \sigma_p = \beta_{OR} \mu b M (f_v)^{1/2} / d_p$$  \hspace{1cm} (2)

with the coefficient $\beta_{OR} = 1/(2\pi) \ln\{d_p[\Pi/(6 f_v)]^{1/2}/b\}$ for screw dislocations. Here, $f_v$ is the volume fraction of uniform distributed particles of mean diameter $d_p$ in parallel glide planes, $\mu$ the shear modulus, $b$ the Burgers vector and $M \approx 2.73$ the Taylor factor describing texture. Dynamic fracture as observed from Charpy-impact tests is assumed to occur stress-induced by micro-crack formation within a local damage area of plastic the crack-zone before the main crack. It instable propagates, if the triaxial stress locally exceeds dynamic fracture stress $\sigma_f$ at strain rates of order $\varepsilon^* = 10^3 \text{ s}^{-1}$. Brittle fracture then appears below the ductile to brittle transition temperature DBTT corresponding to the condition for the uniaxial yield strengths $\sigma_y(T=\text{DBTT}) \geq \sigma_f = \sigma_f / k_1$ where $k_1$ denotes the stress intensity at this local fracture area. Taking into account the temperature ($T$) and strain rate ($\varepsilon^*$) dependence of thermally activable yield strength $\sigma_y(T, \varepsilon^*) = P \exp(-\beta_{d} T)$ [6] together with their athermal component $\sigma_o = \sigma_{dk} + \Delta \sigma_p + \Delta \sigma_i$, the transition temperature DBTT can be expressed by [1,7a]

$$\text{DBTT} = - (1/\beta_{d}) \ln[(\sigma_f^{*} - \sigma_o) / P]$$  \hspace{1cm} (3)

dependent on maximum thermal yield stress $P$ (Peierls stress) at $T=0 \text{ K}$ and the material coefficient $\beta_{d} = \beta_o - \beta_1 \ln \varepsilon^*$ with their constants $\beta_o$, $\beta_1$. For calculations of Ta≤40W, the coefficients $\beta_o=5.35 \times 10^{-3} \text{ K}^{-1}$, $\beta_1=3.27 \times 10^{-4} \text{ K}^{-1}$ [5,6] with $P=1125 \text{ MPa}$ [6,8,9] as observed for tantalum have been used. The strength-induced, relative increase of initial DBTTo according to eq. 3 can be in approximation described by $V=\Delta \text{DBTT}/\text{DBTT}_o \approx (K_o/P)\Delta \sigma_o$ for lower strengths $\Delta \sigma_o$, where the constant $K_d \geq \varepsilon=2.718$ is only weakly dependent on $T$, $\varepsilon^*$. This approximation which gives $K_o/P \approx 0.00278 \text{ MPa}^{-1}$ for the reduced activation ferrite-martensitic (RAFM)-(7-9)CrWVTa steels agree about with the DBTT shift $\Delta \text{DBTT}=K \Delta \sigma_o$, $K \approx 0.6 \text{ MPa}^{-1}$ deduced from 3-dimensional FEM calculations [10] of plasticity within the damage zone of crack for the similar 7CrWVTa(RAFM) steel F82H in dependence of irradiation strengthening $\Delta \sigma_o$, measured at the irradiation equal test temperature of
T_{tr}=T=300°C. Ductile rupture described by the upper shelf energy USE of Charpy-tests is assumed to occur by strain-induced damaging through void formation and their coalescence within the local fracture zone. Fracture appears if the local stress within the critical damage zone achieves the ductile fracture stress \( \sigma_f = \sigma_{fd}/k_I \geq \sigma_{f*} \) or at least a critical fracture strain \( \varepsilon_f \) corresponding to a critical damage. The upper shelf energy \( \text{USE} \propto \varepsilon_f \) is proportional to the deformation work \( \varepsilon_f \) done over the whole volume of plastic damage zone up to fracture appearance. The calculation of \( \varepsilon_f \) takes into account the yield strength dependence of plastic zone size \( r_p \propto 1/\sigma_y^2 \) [11] together with the local work-hardening \( \sigma(\varepsilon) \) and stress intensity within the critical damage zone of instable micro-crack nucleation at position \( x_f \) according to the micro-mechanical deformation model described in [1]. A yield strength increase reduces \( r_p \) and critical distance \( x_f \) of fracture initiation to the main crack tip as well as the stress intensity \( k_I \). From this model, the relative reduction of upper shelf energy \( U_p = \Delta \text{USE}_p/\text{USE}_o \) normalized to the initial value \( \text{USE}_o \) for example due to dispersion strengthening \( \Delta \sigma_p \) can be analytically expressed by

\[
U_p = \Delta \text{USE}_p/\text{USE}_o = 1 - \phi \left[ \frac{1}{1+\Delta \sigma_p/\sigma_y} \right] \left( \frac{\varepsilon_f^p}{\varepsilon_f} \right) C_L
\]

in dependence of main microstructural parameters as \( f_v \) and \( d_p \). The fracture mechanical coefficient \( \phi \approx 1 \) which describes the strength-induced change of plastic strain distribution \( \varepsilon(x) \) can be obtained from comparison of eq.4 with FEM calculations. Here, \( \Delta \sigma_{e,m} \) and \( \Delta \sigma_{e,m}^p \) denote the maximum strain hardening at \( \varepsilon \to \infty \) for the initial \( (f_v=0) \) and dispersion-strengthened state \( f_v, d_p \) and \( \varepsilon_f, \varepsilon_f^p \) the corresponding local fracture strains dependent on the relevant ductile fracture stresses \( \sigma_{fd}^*, \sigma_{fd}^p \) and \( R_m(T, \varepsilon') = R_{m,o} \) [1] the normalized dislocation annihilation coefficient, where mobile dislocations spontaneously annihilated at achieve of the critical capture radius distance \( R \geq b \). At least fracture appears at achieve of a critical fracture strain of \( \varepsilon_f = 0.4 \). According to experimental results observed on various bcc/fcc metals the dependence of annihilation coefficient \( R_m(T, \varepsilon') \) [12] can be phenomenical expressed by

\[
R_m \approx R_{m,o} \left( \varepsilon_{o,s}^*/\varepsilon' \right)^{1/z}
\]

with the corresponding coefficient \( R_{m,o} \) at \( T=0 \) K and the temperature dependent exponent \( z=\mu b^3/(kTg) \) where \( g \approx 0.5-1 \) and \( \varepsilon_{o,s} \approx 10^7 \text{s}^{-1} \) are empirical constants. Coefficient \( R_m \) strongly increases with increasing temperature and decreasing strain rate. Maximum strain hardening \( \Delta \sigma_{e,m} \), true fracture strain \( \varepsilon_f \) and uniform ductility \( \varepsilon_u \), which related to the engineering strain via \( \varepsilon_u = 1 - \exp(-\varepsilon_u) \) are given by [1,7a]

\[
\Delta \sigma_{e,m} = \alpha \mu b M^2/(4\Lambda_o R_m) \{1 + \left[ 1+64 R_m \Lambda_o^2 (b M)^{-1} \right](f_v/d_p) \}^{1/2}
\]

\[
\varepsilon_f = -(1/R_m) \ln \left\{ 1 - R_m(\sigma_{fd}/k_I - \sigma_y)/\sigma_c \right\} \quad \varepsilon_u = -(1/R_m) \ln \left\{ 1 - R_m/(1-m +R_m) [1 - (\sigma_y/\sigma_c)(1-m)] \right\}
\]

with the dislocation strength coefficient \( \alpha \approx 0.1464 \) [1] and the strain rate sensitivity \( 1 \geq m = \delta \ln \sigma_y/\delta \ln \varepsilon = -\beta \geq 0 \) of strength. Further, strength \( \sigma_c = \Delta \sigma_{e,m} R_m \) defines the critical yield strength

\[
\sigma_c^* = \Delta \sigma_{e,m} R_m/(1-m) = \sigma_c/(1-m)
\]
above which uniform strain in tensile tests disappears ($\varepsilon_0 = 0$) due to macroscopic plastic instability according to Hart’s instability concept [14] $\delta \sigma_c(\varepsilon) / \delta \varepsilon = \sigma_c(\varepsilon)(1-m)$, which becomes identical with Considere’s law for $m=0$. Deduced relations in eq. 6a take into account an enhanced dislocation multiplication ($q=1$) with increasing density of stored dislocations $\Lambda \propto 1 / \Lambda \rho^{1/2}$ because they act as strong barriers reducing mean glide distance of moving dislocations according to $\Lambda = \Lambda_0 / (b \rho^{1/2})$ with the structural constant $\Lambda_0 \approx b [1,7a]$. The important critical strength $\sigma_c^*$ corresponds to the maximum attainable ultimate tensile strength for rate sensible metals $m \geq 0$ due to macroscopic plastic instability appearance in tensile tests for the case of condition $q=1$ and uniform plastic deformation. It strongly increases with increasing dispersion hardening and also weaker with increasing shear modulus. As eq. 6a demonstrates, increasing strengthening by solid solution and grain refinement reduces uniform ductility and more pronounced the fracture strain. Dispersion hardening otherwise reduces again fracture strain but increases uniform ductility due to enhanced dislocation multiplication [1] below achieve of a critical yield strength $\sigma_y = \sigma_L$. For bcc metals at very low temperatures <0.3$T_m$ (melting temperature), where glide is mainly controlled by the more heavier movable screw dislocations, their glide distance becomes mainly bounded by the grain size $\Lambda = c d_K$, $c \leq 1$ corresponding to constant dislocation multiplication ($q=0$) because they can easily around sessile dislocations by cross-slip. At higher temperatures edge dislocations then becomes easier movable and can more stronger contribute to dislocation glide. Sessile dislocations act now as strong glide barriers which reduce mean glide distance according to the considered behaviour $q=1$. The critical strength $\sigma_L$ above which uniform strain finally becomes limited by the pronounced strength-induced reduction of fracture strain (i.e. $\varepsilon_\delta \leq \varepsilon_u$) in the case of $q=1$ can be exactly expressed by

$$\sigma_L = [(1 - m + R_m)(\sigma_{f,d}/k_L) - \sigma_c] / R_m$$

(7)

where $k_L$ describes stress intensity at $\sigma_L$ and $\sigma_{f,d}$ the ductile fracture stress. It demonstrates, that such limitation of uniform strain by the drop of fracture strain $\varepsilon_\delta$ giving $\sigma_L \leq (\sigma_{f,d}/k_L)$ only appears if critical strength $\sigma_c^*$ for uniform ductility appearance becomes larger than the fracture stress $\sigma_{f,d}/k_L$. For valid of that condition, strength $\sigma_L \leq (\sigma_{f,d}/k_L)$ reduces with increasing strain rate sensitivity $m$ and strength $\sigma_c$ as given for dispersion-hardened metals, but increases with increasing annihilation coefficient $R_m$ and finally asymptotes to $\sigma_L = (\sigma_{f,d}/k_L)$ for $R_m \rightarrow \infty$.

For the reduction of area to fracture, which relates to the true fracture strain by $Z=\Delta A/A_o = 1 - \exp(-\varepsilon_\delta)$ it results $Z=1 - (1 - R_m(\sigma_{f,d}/k_l) - \sigma_y) / \sigma_c$ from eq. 6a in the case of $q=1$, whereby always $k_l \geq 1$ yields for $\sigma_y < \sigma_{f,d}$ because of $\varepsilon_u < \varepsilon_\delta$. For the to zero extrapolated strength $\sigma_y \rightarrow 0$ like the maximum true uniform strain $\varepsilon_{u,o} = (1/R_m) \ln\{1+R_m/(1-m)\}$ again their maximum arises here described by $Z_c = 1 - (1 - R_m(\sigma_{f,d}/k_l))/\sigma_c$ which becomes 

$$\sigma_y = (\sigma_{f,d}/k_l)/\sigma_c$$

for the low annihilation coefficient $R_m=1$ as might appear at low deformation temperatures. For that case $R_m=1$ additionally fracture strain according to $Z=(\sigma_{f,d}/k_l) / \sigma_y$ reduces linearly with increasing yield strength, more stronger for dispersion-hardened metals with higher $\mu$. It further demonstrates that for $q=1$ compared to constant dislocation multiplication $q=0$ a weaker drop of fracture strain $\varepsilon_\delta$, $Z$ arises with increasing yield strength and maximum work-hardening $\Delta \sigma_{exm}$ due to fine dispersions in contrast to the behaviour of uniform ductility. For $q=0$ it results $\sigma_y \rightarrow \infty$ and uniform strain with the compared to $q=1$ initial lower maximum of $\varepsilon_{u,o} = (1/[2(R_m)]) \ln\{1+R_m/(1-m)\}$ at $\sigma_y = 0$ continuously reduces with increasing yield strength and asymptotes finally to $\varepsilon_u \rightarrow \infty$ at $\sigma_y \rightarrow \infty$. Thus, bcc metals at lower $T$ following the behaviour $q=0$ show higher uniform ductilities at high yield strengths but lower uniform strains at low strengths especially at $\sigma_y = 0$ of their appeared maximum.
For the description of grain refinement and irradiation hardening effects on the change of the ductile impact energy shift $U = \Delta \text{USE}_{dk,i}/\text{USE}_p$ normalized to initial value of dispersion-hardened state $f_v > 0$ again eq. 4 can be used by replacing $\Delta \sigma_p$ through the caused strength increase $\Delta \sigma_{dk}$ or (eq. 1b) $\Delta \sigma_i$. In the case of grain refinement also an increase of fracture strength according to the Hall-Petch type relation

$$\sigma_{fd} = \sigma_{fd,o} + k_f / d_k^{1/2} \tag{9}$$

has been additionally taken into account with the fracture stress coefficient $k_f$ and the fracture stress $\sigma_{fd,o}$ at $d_k \to \infty$. With this, the true fracture strain then can be expressed by

$$\varepsilon_f = -(1/R_m) \ln \left\{ 1 - \left[ \sigma_{fd,o}/ \sigma_o + (k_f - k_{HP}) / d_k^{1/2} \right] R_m / \sigma_c \right\} \tag{10}$$

Accordingly, the strength-induced drop of fracture strain distinctly weakens with increasing constant $k_f$ of fracture stress increase. For the strength dependence of stress intensity at position of micro-crack nucleation the limitation $2.6 \leq k_I \leq 1.8$ with a linear relation $k_I = k_{I0} - c(\sigma_y - B)$ with $B = 147$ MPa for Ta and $B = 500$ MPa for Ta-(20-40)W and W as well as $c = 1.5 \times 10^{-3}$ MPa$^{-1}$, $k_{I0} = 2.6$ has been used. The total normalized shifts in DBTT and ductile impact energy USE according to eqs. 3, 4 are finally given by

$$V_o = \Delta \text{DBTT}/\text{DBTT}_o = V_p + V(1 + V_p) \quad \text{and} \quad U_o = \Delta \text{USE}/\text{USE}_o = U_p + U(1 + U_p) \tag{11}$$

where shifts $V = \Delta \text{DBTT}_{dk}/\text{DBTT}_p$ and $U = \Delta \text{USE}_{dk}/\text{USE}_p$ describing grain refinement effects are normalized to the absolute values DBTT$_p$, USE$_p$ of the dispersion-hardened state $f_v > 0$ at $d_k \geq 20 \mu$m and DBTT$_o$, USE$_o$ are the corresponding initial values at $f_v = 0$. The pure dispersion-induced shifts are denoted by $V_p = \Delta \text{DBTT}_{dk}/\text{DBTT}_o$ and $U_p = \Delta \text{USE}_{dk}/\text{USE}_o$ at the normal grain size $d_k \geq 20 \mu$m.

3. Results
3.1 Constant ductile and dynamic fracture stresses

The main physical properties as Burger`s vector $b$, shear modulus $\mu$, melting temperature $T_m$, as well as thermal conductivity $\lambda_{th}$, linear extension coefficient $\alpha_{cx}$, cross section for fast $^{14}$MeV neutrons and half time of decay of main radioactive isotope are given in Tab.1 for the considered refractory metals Ta, W, Ta-40W and possible alloying elements Mo, Cr, V at room temperature (RT) which compared with $^{\alpha}$-Fe. Accordingly, the more brittle W shows the highest $\mu$, $\lambda_{th}$ and lowest thermal expansion coefficient compared to the ductile metals $^{\alpha}$-Fe and Ta. Thus, alloying of Ta with W similar as by Mo and Cr enhances strength and corrosion resistance and reduces thermal stress generation as well as hydrogen embrittlement [13]. By the little lower half time of radiactive decay for W, additionally the radiation activity is reduced as particularly required for their application as structural materials in nuclear devices. This can be further reduced by an additional alloying of Mo and Cr which reduces $\alpha_{cx}$ and increases $\lambda_{th}$ but also $\mu$. From the model-assisted fit of the stress-strain curves including the initial strain hardening rates, the high critical strength $\sigma_c = 1761$ MPa has been deduced for pure Ta [2] at RT and $\varepsilon^* = 10^{-4}$ s$^{-1}$, $d_k = 20 \mu$m, $\sigma_y = 149.5$ MPa which follow the multiplication behaviour $q = 1$ with the annihilation coefficient $R_m = 6.5$. Critical strength increases by tungsten alloying to $\sigma_c = 2670$ MPa at 20at% W, whereby $R_m = 6.675$ and $\sigma_y = 590$ MPa. These critical strengths of Ta-(0-20)W are higher than these $\sigma_c = 850-950$ MPa obtained for austenitic Cr/Ni stainless steels of types AISI 304/316, DIN 1.49881 [15] at $T_{irr} = T = 230^\circ$C from the drop of uniform ductility by irradiation strengthening due to atomic clusters and dislocation loops formation. A somewhat lower critical strength $\sigma_c = 775$ MPa is also deduced.
from the dependence $\varepsilon_u(\sigma_y)$ due to irradiation strengthening for the 7-9CrWVTa (RAFM) steels (0.07-0.1 wt.% C) developed as structural materials for nuclear applications as the blanket of future fusion reactors. Increasing (C+N) contents through pronounced carbide/nitride precipitations increase according to eqs.6a,b critical strength $\sigma_c$ of conventional 10CrMoVNb steels to slightly higher values of $\sigma_c \approx 1150$ MPa as found in the 10CrMoVNb steel MANET-I with 0.14 wt.% C. Thus, the high critical strength values $\sigma_c=1761-2670$ MPa of Ta-(0-40)W alloys corresponding to $\sigma_c^*$ for $m=0$ might indicate that dislocation multiplication results here by reactions of mobile with stored dislocations within the matrix and partly also by grain boundaries. Fig. 1 at first shows the observed yield strength increase for various ferritic and ferritic martensitic ODS-(7-13)CrWVTa-(0.1-0.5 wt.% yttria) steels produced by powder metallurgy as a function of the dispersion parameter $(f_v)^{1/2}/d_p$, which compared with the Orowan predictions using $\alpha=0.1464$, $M=2.733$, $b=0.25$ nm and $\mu=82.3\text{GPa}$ [1]. The particle strengths 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 $f_v=1.5618\times10^{-2}$×(wt.% yttria). As demonstrated, the measured strengths $\Delta\sigma_p \leq 970$ MPa at the particle sizes $d_p \geq 3$ nm increases with increasing ratio $(f_v)^{1/2}/d_p$ consistently with the Orowan predictions for uniform distributed particles. The deviation of measurements toward lower strengths mainly result from softening due to the ferritic-martensitic duplex structure of ODS-steels compared to fully martensitic base steels or non-uniform particle distributions. Thus, the Orowan predictions seem to be an upper limit of achievable strengthening by oxide dispersions and non-shearable precipitates. Mainly due to the higher shear moduli a stronger dispersion hardening can by observed in ODS-Ta-W alloys at higher tungsten contents above 11 at. W % compared to ODS-(7-13)Cr-RAFM steels at the same particle factors $f_v$, $d_p$ and uniform distributions. It achieves with an increase of factor ~2.1 their maximum for pure ODS-W. For the low particle fraction $f_v=0.00469$ corresponding to 0.3wt.% $Y_2O_3$ for ODS-RAFM and the mean particle size $d_p=12$ nm, already high dispersion strengths of $\Delta\sigma_p \approx 352.9$ MPa - ODS-Ta-20W, $\Delta\sigma_p \approx 411.03$ MPa - ODS-Ta-40W and $\Delta\sigma_p \approx 670.19$ MPa for ODS-W are obtained compared to $\Delta\sigma_p \approx 318.13$ MPa for ODS-RAFM and $\Delta\sigma_p \approx 267.4$ MPa for ODS-Ta. Fig. 2a shows the predicted grain size dependence of engineering uniform ductility $\varepsilon_u$ vs. $d_K^{-1/2}$ for ODS-RAFM steels at RT and $d_p=12$ nm, which following the work-hardening behaviour $q=0$ as function of $f_v$ (0 to 0.03). The following parameters have been used for calculations: $k_{HP}=6641$ MPa(nm)$^{1/2}$, $\mu=83.3$ GPa, $\sigma_{fd}=2100$ MPa and coefficient $R_m=6$ which predicts a maximum uniform strain of $\varepsilon_{u,0,*}=0.150$ at $m=0$. For the given martensitic structure with lath thickness of ~300 nm at $d_K=20$ nm it is assumed that parameter $c \leq 10$ decreases with decreasing grain size and achieves $c=1$ for $d_K \leq 10$ nm. For lower particle fractions $f_v \leq 0.05$, most pronounced at $f_v=0$, uniform ductility continuously increases by grain refinement and tends to saturate at the value $\varepsilon_u,sat=0.116$ for nc sizes $d_K \to 0$. In accordance with predictions, this saturation ductility, which for low $\varepsilon_{u,sat}$ or $\mu/k_{HP}$ values can expressed by the simpler upper limiting approximations

$$\varepsilon_{u,sat} \leq (\alpha \mu/k_{HP})^2 \beta M^3 /[4 (c(1-m)^2)] \leq \varepsilon_{u,0,*}$$

(12)

becomes independent on volume fraction $f_v$ and solid solution hardening. It increases with increasing $m$ and ratio $(\mu/k_{HP})^2$ but also with increasing texture factor $\alpha M^3$. Toward higher $\varepsilon_{u,sat}$ or lower $\mu/k_{HP} \to 0$ values, this increase of saturation ductility $\varepsilon_{u,sat}$ weakens and finally approaches the maximum value $\varepsilon_{u,sat} \approx (1/R_m) \ln [1+R_m/(1-m)]$ which depends besides $R_m$ also on $m$. This demonstrates, that $\varepsilon_{u,sat}$, similar as the maximum ductility $\varepsilon_{u,0,*}$ now increases with decreasing annihilation coefficient $R_m$ and weaker also with increasing $m$ but becomes independent on ratio $(\alpha\mu/k_{HP})$. That deduced saturation ductility generally becomes valid also for bcc/fcc metals which
initial following the behaviour $q=1$ because dislocation multiplication at nc region becomes finally determined by the grain size ($q=0$). 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 $f_v\leq0.024$ at larger $d_K$ but decreases weakly at lower $d_K$. However, when a lower critical grain size $d_{K,c}\geq90\text{ nm}$ is achieved within the uf region corresponding to $\sigma_y\geq\sigma_{L}$, fracture strain drastically drops and limits uniform ductility by $\varepsilon_f<\varepsilon_u$. Finally, it disappears at the nc grain size $d_{K,f}$, where $\sigma_y=\sigma_{fd}$. With increasing fraction $f_v$ and decreasing $d_{K}$, the limiting grain size $d_{K,c}$ for a uniform ductility increase increases more strongly than $d_{K,f}$. At higher particle fractions, as $f_v\geq0.024$ in the case of $d_p=12\text{ nm}$, uniform ductility then becomes always limited by $\varepsilon_f<\varepsilon_u$. Fig. 2b shows the predicted grain size dependence $\varepsilon_{u,n}$ vs. $d_{K}^{-1/2}$ now for ODS-Ta again at RT-$q=1$ as function of $f_v$ for $d_p=12\text{ nm}$ and for different particle sizes $d_p$ at $f_v=0.00469$. Additionally, the effect of an irradiation hardening of $\Delta\sigma_i=600\text{ MPa}$ is indicated. The following parameters have been used for calculations: $k_{HP}=7273.5\text{ MPa(nm)}^{1/2}$, $\mu=61.8\text{ GPa}$, $c=1$ with the ductile fracture stress $\sigma_{fd}=2100\text{ MPa}$. Contrary to ODS-RAFM at RT-$q=0$, the initial quite higher uniform ductility with the maximum $\varepsilon_{u,0}=0.310$ for Ta and ODS-Ta shows a continuous decrease from grain refinement, more strongly at lower $d_K$ and would disappear in the case of $f_v=0$ below $d_{K,c}=123\text{ nm}$ up to $d_{K,f}=39\text{ nm}$. It shows a qualitative similar behaviour as predicted for ODS-RAFM steels at the higher $T=300\text{ °C}$, where the multiplication process change from $q=0$ at RT to $q=1$ because edge dislocations mainly control deformation [1]. However, for both RAFM and Ta at $f_v=0$, a transition from $q=1$ to $q=0$ occurs at lower grain sizes, which about $\varepsilon_{u,min}\approx0.1$ for Ta because glide becomes then limited by the grain size $\Lambda=c\ d_K$. By this, uniform ductility then increases again from the minimum toward to a saturation value, which with $\varepsilon_{u,sat}=0.106$ for ODS-Ta is somewhat higher than $\varepsilon_{u,sat}=0.0719$ of ODS-RAFM steels at $300\text{ °C}$. For both ODS-Ta and ODS-RAFM otherwise at higher fractions $f_v\geq0.00469$ and lower $d_p\leq50\text{ nm}$, no such transition $q=1\rightarrow q=0$ appears because glide remains always limited by the high dislocation density up to fracture. With increasing fraction $f_v$ (0 to 0.03) as shown in Fig. 2b for $d_p=12\text{ nm}$, uniform ductility of ODS-Ta increases also but somewhat more weakly at lower $d_K$ due to the combined strong increase of critical strength $\sigma_c$. However, critical grain size $d_{K,c}$ strongly increases by increasing $f_v$ respectively dispersion hardening. With decreasing $d_{p}$ as shown for $f_v=0.01$, $\varepsilon_u$ is reduced more pronounced for lower $d_p\leq5\text{ nm}$, where uniform ductility becomes finally limited by the drop of fracture strain. Irradiation hardening of $\Delta\sigma_i=600\text{ MPa}$ strongly decreases $\varepsilon_u$ and increases $d_{K,f}$. A similar behaviour $\varepsilon_u$ vs. $d_{K}^{-1/2}$ as Fig. 2c demonstrates with, however, lower ductilities occurs also for the higher W containing Ta-20W alloy ($f_v=0$) showing a lower $\varepsilon_{u,min}=0.085$ but somewhat larger $\varepsilon_{u,sat}=0.11$ and $d_{K,f}$ compared to pure Ta. For ODS-Ta-20W with $f_v=0.01$ and $d_p=12\text{ nm}$, uniform ductility increases at $d_K=100\text{ nm}$ but reduces below that earlier because transition $q=1\rightarrow q=0$ is suppressed by their larger $d_{K,f}$ values.

As already shown for ODS-RAFM steels in ref. [1], dispersion hardening increases uniform ductility below achieve of the critical strength $\sigma_y\leq\sigma_L$. Interestingly, maximum uniform ductility increase is attained at an optimum particle size $d_{p}^*$, which can be analytically expressed in the case of constant multiplication $q=0$ as expected for bcc metals at lower temperatures by [1]

$$d_{p}^* = c\ d_K \ f_v \{(\sigma_o + \sigma_{dk}* c) \ d_K \ f_v^{1/2}/(\beta OR \mu b M) - 1 \}^{-1}$$

(13a)

where $\sigma_{dk}* =k_{HP} \ d_{K}^{-1/2}$. This optimum particle size, which for ODS-RAFM steels at RT-$q=0$ is at nc scale $d_{p}^*=7-22\text{ nm}$ increases with increasing volume fraction $f_v$, $\mu$ and Taylor factor $M$ but reduces with increasing strengthening by solid solution, irradiation defects and grain refinement particularly at larger constants $k_{HP}$. Further, $d_{p}^*$ remains independent on annihilation coefficient $R_m$.
and strain rate sensitivity $m$ whereby, however, the maximum attainable ductility increase at $d_p^*$ increases with increasing $m$, initial strength $\sigma_{o,m} + \Delta \sigma_i$ and coefficient $k_{HP}$ but reduces with increasing shear modulus. Also for enhanced multiplication $q=1$ again such optimum particle size $d_p^*$ appears. For higher strengths this can be analytic expressed by

$$(d_p^*)_{q=1} = \beta_{OR} \mu' b M f_v ^{1/2} \{[\sigma_o + \sigma_{dk}^*] / (\sigma_o + \sigma_{dk}^* - 1/\sigma_o^2)\} \Omega d_p^{-1} \}$$

with the corresponding optimum particle hardening $\Delta \sigma_p^* = 2(\sigma_o + \sigma_{dk}^*) / (\sigma_o + \sigma_{dk}^* - 1/\sigma_o^2)\} \Omega d_p^{-1}$ and $\Omega = 64 R_m (\Lambda_o / (bM))^2 \{[(\beta_{OR} \mu')^2]$, which similar to eq. 12a. Compared to $q=0$, optimum particle size $d_p^*$ increases for $q=1$ little weaker with increasing $f_v$ (about $d_p^* f_v ^{1/2}$) but reduces more strongly with increasing strengthening (weaker than $d_p^* \sim 1/\sigma_o^2$) as due to grain refinement or irradiation defect formation. It remains again independent on $m$ as for $q=0$, but additionally becomes now dependent on $R_m$. Increasing annihilation coefficients $R_m$ reduces $d_p^*$ and the corresponding optimum particle strengthening $\Delta \sigma_p^*$. For ODS-Ta by the lower strength and shear modulus slightly higher $d_p^* \geq 30$ nm values at $f_v < 0.05$ arise which, however, strongly reduce with increasing W alloying. It finally asymptotes to $d_p^* \leq 6$ nm for pure ODS-W and $d_p \leq 20$ nm. Thus, increasing tungsten alloying of ODS-Ta-W metals indirectly increases uniform ductility because their optimum shifts to higher particle strengthening. Increasing particle strengthening, however, reduces $\sigma_L$ of condition $\varepsilon_f < \varepsilon_u$ which might limit this increase particularly in the case of lower fracture stresses respectively brittle metals. Fig. 3 finally shows the phase diagram of grain size vs. particle size for appearance of such increase of uniform ductility by grain refinement and dispersion hardening (DIGD) in ODS-Ta and ODS-Ta-20W alloys at $f_v = 0.01$. With decreasing particle size and increasing W content the grain refinement must be limited clear above $d_k \approx 10$ nm to achieve DIGD. As visible, the line $d_k,c$ for $\sigma = \sigma_L$ drops more strongly with decreasing particle size than $d_k,l$ of $\sigma = \sigma_{GL}$.

As shown in [12] the fracture strain of various 9-12CrMo(W)VNb steels at RT denoted by the reduction of area to fracture continuously decreases with increasing strengthening by irradiation defects. Strengthening due to fine Y$_2$O$_3$ dispersions as observed in ODS-7CrWVTa-Eurofer’97 and the ferritic ODS-20CrAIeTiC steel P2000 at 300°C enhances this fracture strain drop by pronounced work-hardening particularly at lower $R_m$ values. At the precipitation hardened Al alloy AA611 [15] at RT by their low $R_m$ and m values due to the low stacking fault energy indeed a linear decrease of fracture strain $Z(\sigma_f)$ with increasing strengthening is observed as predicted for $R_m = 1$ at $q=1$. Fracture stress with values of $\sigma_{f,d} / k_{I,f} = 375-500$ MPa and also the maximum fracture strain $Z_o = 0.55-1$ strongly depends of the ageing temperature $T_a = RT-200°C$. Mean ageing temperatures $T_a = 100-177°C$ with low precipitation sizes increase ductile fracture stress $\sigma_{f,d} / k_{I,f}$ but reduce $Z_o$ to about $\approx 0.55$, while at the highest temperature $T_a = 200°C$ indicating over ageing, the maximum $Z_o = 1$ results. This behaviour will be determined by the superimposed effects from alloy composition and ageing treatment upon precipitation morphology parameters $f_v$, $d_p$ and strain rate and strain depending work-hardening coefficients $m = \delta \ln \sigma_f / \delta \ln \varepsilon$ and $n = \delta \ln \sigma_f / \delta \ln \varepsilon$ through variation of solid solution hardening. It determines $Z_o(m)$ and also the critical damaging $\sigma_{f,d}(f_v)$ by void formation at precipitates. These observations demonstrate that also in dispersion-hardened ODS-metals as ODS-RAFM steels and ODS-Ta-W alloys besides the pronounced strengthening additionally also a change in fracture stress might occur due to present fine precipitates or dispersions. Fig.4a shows the effect of grain refinement on the DBTT of ODS-Ta alloys as predicted by eq. 2,3 in dependence of particle size $d_p = 4-30$ nm at constant fraction $f_v = 0.00469$ and dynamic fracture stress $\sigma_f = \gamma \sigma_{GL}$ with $\sigma_{GL} = 2400$ MPa and $r_f = 0.9$. Accordingly, DBTT increases by grain refinement more pronounced within the nc region and at lower particle sizes respectively stronger dispersion hardening. This increase within the uf grain size region of $d_k \geq 100$ nm remains, however,
comparably weaker. Additionally in Fig.4b, the critical grain size $d_{k,c}$ where DBTT achieves RT is plotted for ODS-Ta and ODS-Ta-20W alloys as function of particle volume fraction at $d_p=12$ nm. This limiting grain size is particularly important for alloy optimisation if excellent impact properties are required at and above RT. As shown, $d_{k,c}\geq50$ nm progressively increases with increasing particle fraction and achieves the uf size of 80 nm at $f_i=0.00469$ for ODS-Ta. Changes of the ductile fracture stress to 2100 and 2600 MPa at $f_i=0.00469$ give rise to a critical grain size change of about $+\Delta d_{k,c}\approx30$ nm and $-\Delta d_{k,c}\approx20$ nm respectively. Increasing W alloying of ODS-Ta-W shifts relation $d_{k,c}(f_i)$ due to solid solution hardening to higher critical grain sizes. For ODS-Ta-20W where $d_{k,c}\geq120$ nm it achieves already about $d_{k,c}\approx1050$ nm at $f_i=0.00469$. The correlation between DBTT and uniform ductility of ODS-Ta as calculated by eqs.3,6a for $m=0$ is shown in Fig. 5a in dependence of grain refinement and dispersion hardening through variation of the particle size $4\leq d_p\leq30$ nm at $f_i=0.00469$. As demonstrated, DBTT continuously increases with decreasing uniform strain in first order about along a master curve, more pronounced at lower $d_p$ and higher DBTT values. It indicates that DBTT and uniform ductility are equivalent parameters due to their strong dependency on yield strength, which more pronounced for DBTT compared to $\varepsilon_u$. Thus, grain refinement reduces uniform ductility and increases distinctly DBTT more stronger at lower $d_p$. For particle sizes below $d_p<8$ nm at $f_i=0.00469$, the uniform ductility decrease then becomes more enhanced due to its limitation ($\varepsilon_f<\varepsilon_u$) by the strong reduction of fracture strain at high yield strengths above the critical value $\sigma_L$ as also shown in Fig. 5a by the dotted line. At high DBTT and strength values this limitation shifts the dependence DBTT vs. $\varepsilon_u$ to lower uniform ductilities. The ($-\cdot-\cdot$) lines otherwise denotes the maximum achievable uniform ductility for high ductile fracture stresses without their limitation by the fracture strain (i.e. $\varepsilon_f<\varepsilon_u$). A reduction of fracture stresses at constant ratio $r_f=\sigma_f/\sigma_{fd}=0.9$ increases DBTT and reduces also strongly uniform ductility because it becomes more stronger limited by the lower fracture strain via a decrease of $\sigma_L$. Thus, the dependence DBTT($\varepsilon_u$) together with their achievable maximum DBTT($\varepsilon_u,max$) for higher fracture stresses $\sigma_{fd}$ shift to lower uniform ductilities especially at lower DBTT values. The increase in DBTT by dispersion hardening can be approximated by $V_p=\Delta DBTT_p/DBTT_{c=2.416}\cdot10^{-3}\Delta \sigma_p$. Grain refinement increases DBTT and reduces USE, thus, degrades impact properties. The effect of grain refinement on the correlation between DBTT and the ductile upper shelf energy shift $U=\Delta USE_{dk}/USE_p$ of ODS-Ta is shown in Fig. 5b as function of volume fraction $f_i=0-0.03$ at $d_p=12$ nm and $r_f=0.9$. Also lines for constant grain sizes are indicated in that plot. As demonstrated, a grain refinement increases initial weaker DBTT but more stronger the ductile energy reduction $U$. At lower grain sizes then the DBTT increase enhances and shift $U$ asymptotes quickly to $U\rightarrow1$ according to $USE\rightarrow0$. Increasing particle volume fraction increases further the initial DBTT value at larger $d_k$ and additionally enhances the grain refinement-induced increase of DBTT and shift $U$ particularly at low grain sizes $\geq0.25\ \mu m$ of uf and nc regions.

3.2 Effect of a grain refinement-induced increase of ductile/dynamic fracture stresses $c_f>0$

Fig. 6a shows the effect of a grain refinement-induced increase of fracture stresses denoted by the ratio $1.5\leq c_f=k_f/k_{HP}\geq0$ on the correlation between DBTT and uniform ductility of ODS-Ta alloys through grain refinement at $f_i=0.00469$, $d_p=12$ nm, $\sigma_{fd,o}=2400$ MPa and $d_k=20\ \mu m$, $\varepsilon_f=0.4$. Most interestingly, a minimum in DBTT within the uf region $d_{k,min}\approx100-200$ nm results which becomes more pronounced at higher ratios $c_f\geq0.3$ respectively for a stronger fracture stress increase at constant $r_f=0.9$. In pure Ta that minimum appears at comparably lower grain sizes 30–70 nm within the nc region. That grain size at DBTT minimum together with its DBTT$_{min}$ value increase with pronouncing dispersion hardening. Besides, increasing ratio $c_f$ shifts the region of boundary condition $\varepsilon_f<\varepsilon_u$ to lower grain sizes and higher DBTT values promoting an additional uniform
ductility increase. Furthermore in Fig. 6b, the critical grain sizes $d_{k,c}$ for ODS-Ta, Ta-W and ODS-Ta, ODS-Ta-20W alloys are plotted in dependence of ratio $c_f=k_f/k_{HP}$ at $f_v=0.00469$ and $d_p=12$ nm. It progressively decreases with increasing ratio $c_f\leq1.5$ for the Ta, Ta-20W and ODS-Ta alloys but distinctly weaker at the higher-strengthened ODS-Ta-20W. For pure Ta the obtained critical grain sizes $d_{k,c}=3-33$ nm are within the lower bond of the nc region and shift by pronounced dispersion strengthening for ODS-Ta to $d_{k,c}=8.25-76$ nm of the upper bond of nc region. As shown for Ta also the grain size $d_{K,min}$ at minimum DBTT$_{min}$ decreases weakly with increasing ratio $c_f$. Pronounced solid solution hardening of the Ta-20W alloy again shifts relation $d_{k,c}(f_v)$ to higher $d_{k,c}\leq155$ nm which reaches already the uf region. At ODS-Ta-20W then a distinctly stronger increase of critical grain size to $0.8\leq d_{k,c}\leq17.5$ $\mu$m arises for $c_f\leq1.5$ by additionally weakening the dependence $d_{k,c}(c_f)$.

Figs. 7a,b show the effect of a grain refinement-induced increase of dynamic and ductile fracture stresses described by the ratio $c_f=k_f/k_{HP}>0$ on the correlation between DBTT and the ductile energy reduction $U$ of ODS-Ta (a) and Ta-20W(b) alloys due to grain refinement. As indicated for ODS-Ta at $f_v=0.00469$, $d_p=12$ nm increasing $c_f$ reduces stronger DBTT and weaker energy shift $U$. Besides, again a distinct minimum in DBTT of 60–120 K appears around the nc grain size of $d_{K,min}=85$ nm for $c_f\geq0.5$ which pronounces at higher $c_f$ values by decreasing the DBTT$_{min}$ and weaker also $d_{K,min}$. For grain sizes below $d_{K,min}$ both DBTT and shift $U$ are strongly increased. At the Ta-20W alloy this minimum of DBTT$_{min}=110$ K becomes broader and appears at slightly larger $d_{K,min}=150$ nm values (Fig.5b) than for pure Ta. Additionally, at higher ratios $c_f\geq0.8$ and grain sizes $d_k\geq200$ nm the ductile energy shift $U$ becomes negative at a certain higher optimum grain size $d_{k,min}=300$ nm as for the minimum DBTT$_{min}$ corresponding to an increase of USE. Fig. 8a shows the grain size dependence of DBTT for the higher tungsten containing Ta-40W alloy at $5\geq c_f\geq0$. Accordingly, the DBTT increases by grain refinement from initial value 153 K for the normal grain size $d_k=20$ $\mu$m, more pronounced below $d_k\leq400$ nm of uf grain sizes. For a mean fracture stress increase with ratios $0.3\leq c_f\leq2$, the DBTT$_{min}$ appears at the optimum grain size of 450-500 nm and toward lower $d_k$, then again DBTT strongly increases. At high ratios $c_f\geq1.5$, then DBTT continuously decreases also within the nc region while energy shift $U$ is increased after going through its minimum. This demonstrates Fig.8b where the map of relative shifts $V_o=\Delta$DBTT$_{dk}$/DBTT$_{o}$ vs. $U_o=\Delta$USE$_{dk}$/USE$_{o}$ is shown in dependence of grain refinement for various $c_f=0.5$ to 5 values. At lower $c_f\leq1$, maximum reductions of $V_o$ and $U_o$ are achieved by grain refinement at somewhat higher $d_{K,min}=600$ nm. Toward the nc regime shifts $V_o$ and $U_o$ strongly increase where fracture becomes strain controlled at achieve of the critical strain $\varepsilon_f=0.4$. The behaviour $V$ vs. $U_p=\Delta$USE$_{dk}$/USE$_p$ for ODS-Ta-40W is shown in Fig. 9 for $f_v=0.001$, $d_p=12$ nm. For low $c_f\leq1.5$, the initial higher $V_p=0.247$ and $U_p>0$ values of dispersion-strengthened ODS-Ta-40W compared to the base alloy Ta-40W continuously increase further by grain refinement. At the higher $c_f=2.5$ value then shift $V$ decreases up to the nc region while $U_p$ becomes negative corresponding to a decrease of impact energy USE and goes through a sharp minimum around $d_{K,min}=100$ nm after which it again increases. It becomes then positive at the nc region while shift $V$ still further reduces due to decrease of DBTT. Thus, increasing tungsten alloying of ODS-Ta-W restricts the applicable grain refinement if only a weak fracture stress increase occur and if low DBTT$\leq$RT values are required but additionally enhances strength and more stronger their hydrogen embrittlement resistance. Finally Fig. 10 shows the predicted grain refinement effects of pure tungsten at RT in dependence of ratio $1.5\leq c_f \leq5$ of fracture stress increase which compared with experimental results reported by [4]. Following parameters have been used for predictions: $R_m=4.5$, $k_{HP}=7273.24$ MPa(nm)$^{1/2}$, $\mu=160$ GPa, $\sigma=725$ MPa, $r_f=0.9$, $\beta_m=5.35\times10^{-3}$ K$^{-1}$, $\beta_1=-1.09\times10^{-4}$ K$^{-1}$, P$=1125$ MPa, as well as $\sigma=2670$ MPa, $\sigma_{fd,o}=1600$ MPa and $R_m=4.5$. It predicts a maximum attainable uniform ductility of $\varepsilon_{u,o}=0.315$ which, however, due to the low fracture stress generally limited by $\varepsilon_{f} < \varepsilon_{u}$. The measured DBTT values strongly drops from ~542 K at $d_k=120$ $\mu$m to ~300 K at the uf grain size of $d_k=1000$ nm. Toward higher
grain sizes 1000 >d_K>120 µm again DBTT decreases to ~450 K. At the mean ratio c_f=1.5, the model predicts a minimum of DBTT_{min} ≃ 400 K at d_K,min= 100 nm below which it strongly increases. At higher ratios c_f=2.5/ 5, the grain refinement, however, continuously reduces DBTT. If then fracture stress below d_K= 500 nm remains constant, the DBTT would again strongly increase as denoted by the -.- dotted lines. The measured drop in DBTT agree about with predictions for an initial strong fracture stress increase via the ratio c_f=5 up to about d_K= 1000 nm. The measured DBTT reduction otherwise at high grain sizes above d_K ≃ 120 µm results due to softening by recrystallization of about ∆σ_o ≃ 98 MPa. By grain refinement of the carbide (TiC, Mo_2C) dispersed W-0.3TiC-1.7Mo alloy [17] produced by mechanical alloying following sintering and hot working again such strong increase of fracture stress up to about σ_f ≃ 2400 MPa at d_K=500-1150 nm have been observed with TiC, Mo_2C precipitates of nc sizes 10-25 nm. Thus, grain refinement particularly to the uf region of 100-1000 nm offers the possibility for ductilisation of usual brittle metals such as W, Cr, Mo and strong dispersion-hardened ODS-RAFM and ODS-Ta-W alloys by a possibly resulting strong increase of fracture stresses due to change of fracture process.

4. Conclusions and summary

The following can summarize the main results of this work:
A strong dispersion hardening via the Orowan process increases uniform ductility of ODS-Ta-W alloys like generally of bcc and fcc metals whereas it is reduced by solid solution, grain boundary and irradiation strengthening. Such paradoxes behaviour of enhanced ductility by increasing strengthening has been observed in ferritic-martensitic ODS-(7-13)Cr steels [1] and uf grained AL [18] produced by ECAP. The uniform ductility maximum is achieved at a nano-scaled optimum particle size d_p* which reduces with increasing matrix strength, grain refinement and shear modulus as well as decreasing particle fraction but remains independent of strain rate sensitivity m of strength. For martensitic ODS-(7-13)CrW(Mo)VTa(Nb) steels the optimum particle size achieves d_p* ≃ 7-22 nm at RT for f_p<0.05. For pure ODS-Ta a certain larger optimum particle size d_p* ≃ 30-40 nm arises which strongly reduce with increasing W alloying and achieves finally d_p* ≤ 5 nm for pure ODS-W at d_K ≤ 20 µm. In ODS-W alloys a larger uniform ductility increase might be achieved particularly in the case of a strong dispersion strengthening which, however, becomes limited by the usual lower fractures stresses in these materials which easily leads to appearance of condition σ_y < σ_L already at larger grain sizes. Grain refinement to the uf scale as observed in pure W increases dynamic and ductile fracture stresses and, thus, increases uniform ductility and reduces DBTT. It is also expected that such increase of fracture stresses might occur in Ta-W alloys which would additionally enhance their ductility. For Ta-W alloys at lower W contents the larger optimum particle sizes <40 nm might be achieved also by precipitation reactions from usual fabrication process by the melting reducing production costs.
Grain refinement of Ta-W alloys increases DBTT and reduces upper shelf energy USE as well as uniform ductility in the case of constant ductile/dynamic fracture stresses. By their dominant strength dependences both DBTT and uniform ductility correlate, whereby DBTT always strongly increases with a reduction of uniform ductility either by grain refinement or solid solution strengthening and cold working. Dispersion hardening of ODS-Ta-W alloys increases further DBTT and reduces USE. At a very high total strength, especially of a strong dispersion strengthening additionally now uniform ductility might be strongly reduce particularly at lower ductile fracture stresses and grain sizes by their limitation due to the pronounced drop of fracture strain (i.e. ε_f < ε_u) at σ_y < σ_L.
A combined strong increase of dynamic/ductile fracture stresses by grain refinement denoted by the ratio c_f = k_f/k_HP at r_f=const., however, firstly reduces strongly DBTT and weaker also impact energy shift U of (ODS)-Ta-W alloys. Minima in DBTT_{min} and energy shift U_{min} result at different
optimum grain sizes already at mean ratios $c_f \geq 0.3$ of fracture stress increases. Toward nc sizes again DBTT and shift U are increased. These optimum grain sizes which for Ta, ODS-Ta are within the uf region of $d_{k,\text{min}} \approx 50–200$ nm increase with increasing W content. Besides, also the critical grain size $d_{k,c}$, where DBTT achieves RT is increased by increasing W content, more pronounced for ODS-(20-40)W alloys at low ratios $c_f$. It restricts the suitable amount of grain refinement and dispersion hardening for their low temperature application if DBTT<RT is required. Dependent on grain size and dispersion hardening and fracture stress increase ratio $c_f$ the optimum W content of ductile ODS-Ta-W alloys for their high temperature application are between 20 to $\leq 40$ at% W, where indeed hydrogen embrittlement already disappear [13] by an additional increase of corrosion resistance. A higher W content within this region in combination with a medium dispersion hardening is favourable for nuclear application up to $<1300°C$ where sufficient RT-ductility is required. Grain refinement with a combined increase of fracture stresses can further strongly improve embrittlement resistance of these alloys as found in uf grained W alloys and additionally increases uniform ductility by an increase of fracture strain with more stronger suppress of appearance of condition $\varepsilon_f < \varepsilon_u$ at $\sigma_f < \sigma_L$.

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Fig. 10: Comparison of measured grain size dependence of pure tungsten with predictions - Effect of the fracture stress increase ratio $5 \geq c_f=k_f/k_{HP} \geq 0$.

Tables

Tab. 1: Main physical properties of Ta, W, Ta-40at% W and Mo, Cr which compared with $\alpha$-Fe. (d.. days, h.. hours, $\Theta$..cross section for $^{14}$MeV neutrons)
Fig. 1

![Graph showing Orowan predictions.](image-url)

- ODS-Eurofer, FZK [5], PSI [6]
- ODS-8Cr(0.1-0.3)Ti-(1-3)W, \( d_p = 7-10 \text{ nm} \), JARI [3]
- ODS-8Cr(0.1-0.3)Ti, \( d_p = 12 \text{ nm} \), 0.1-0.3% yttria, JARI [3]
- K5, K2, B.N. Goshchitskii [7]
- ODS-12y1, R.L. Klueh [4]
- 0.25% yttria
- C.Q. Chen [8]
Fig. 2a

ODS-RAFM
RT, $R_m = 6, \sigma_{td} = 2100$ MPa
$k_{ip} = 6641$ MPa(nm)$^{1/2}$

$\varepsilon_{u,sat}$

$d_K,c$

$d_K,f$

$\varepsilon_{u,n}$

$f_V=0$

$d_p=12$ nm

$\varepsilon = \varepsilon_f$

$0.03$

$0.01$

$0.005$

$0.005$

$0.01$

$0.02$

$0.03$

Uniform ductility $\varepsilon_{u,n}$

$d_k^{-1/2}$ (nm$^{-1/2}$)

$d_p$ (nm)

$10^{-3}$ $10^{-2}$ $10^{-1}$ $10^0$ $10^1$
Fig. 2b

ODS - Ta
300K, 10^{-5} s^{-1}, R_m=6.675
\sigma_e=1761 MPa
\sigma_{\text{f,a}}=2100 MPa
k_{\text{II}}=7273.2 MPa(nm)^{1/2}

- f_r=0.01
- d_p=5 nm
- d_p=3.1 nm
- f_r=0.03, d_p=12 nm

Uniform ductility \( \varepsilon_u \)

- d_K^{-1/2} (nm^{-1/2})

Fig. 2c

ODS - Ta - 20W
300K, 10^{-5} s^{-1}, R_m=6.675
\sigma_e=2670 MPa, \sigma_{\text{f,a}}=2100 MPa
k_{\text{II}}=7273.2 MPa(nm)^{1/2}

- f_r=0.01
- f_r=0
- d_p=50 nm
- d_p=500 nm
- d_p=12 nm

Uniform ductility \( \varepsilon_u \)

- \varepsilon_u = \varepsilon_f

- d_K^{-1/2} (nm^{-1/2})
Fig. 3

Graph showing the relationship between grain size and particle size for ODS-Ta and ODS-Ta-20W materials. The graph indicates the brittle region, ductile DIGD region, and the particle size distribution.

Fig. 4a

Graph showing the effect of grain size on DBTT (Ductile to Brittle Transition Temperature) for ODS-Ta material. The graph includes lines for different particle sizes (4 nm, 6 nm, 8 nm, and d_p=30 nm).
Fig. 4b

ODS - Ta - 20W

\[ \sigma_{\text{f}} = 2100 \text{ MPa} \]

ODS - Ta

\[ \sigma_{\text{f}} = 2600 \text{ MPa} \]

ODS - Ta - W

\[ \sigma_{\text{f}} = 2400 \text{ MPa}, \ v_{\text{f},0} = 0.4 \]

\[ d_p = 12 \text{ nm} \]

Volume content \( f_v \)
Fig. 5a

ODS - Ta
\( f_r = 0.00469, \ R_m = 6.6 \)
\( \sigma_{\text{fct}} = 2400 \text{ MPa}, \ \varepsilon_{\text{fct}} = 0.4 \)

- \( \varepsilon_u \leq \varepsilon_f \)
- \( \varepsilon_u \geq \varepsilon_f \)
- \( \varepsilon_{u,\text{max}} \)

\( d_p = 30 \text{ nm}, 250 \text{ nm} \)
\( d_k = 25 \mu m \)

Fig. 5b

ODS - Ta
\( \sigma_{\text{fct}} = 2400 \text{ MPa}, \ d_p = 12 \text{ nm} \)
\( \varepsilon_{\text{fct}} = 0.4, \ r = 0.9, \ R_m = 6.52 \)

- 0.03
- 0.01
- 0.001
- 0.0001
- 0.25
- 0.5
- 1.25

\( f_r = 0, \ d_k = 2.5 \mu m \)

\( U = \Delta \text{USE/USE}_p \)
Fig. 6a

Fig. 6b
Fig. 7a

ODS - Ta
$f_i=0.00469$, $d_p=12$ nm
$\sigma_{f0}$=2400 MPa, $\epsilon_{f0}=0.4$
$r_f=0.9$, $R_m=6.52$

Fig. 7b

Ta-20W
$\sigma_{f0}=2400$ MPa, $\epsilon_{f0}=0.4$
$R_m=6.67$, $r_f=0.9$
Fig. 8a

Ta-40W
\( \sigma_{f0}^* = 971 \text{ MPa, } \sigma_{w0} = 750 \text{ MPa} \)
\( \varepsilon_{f0} = 0.4, 1.8 \leq k_i \leq 2.6 \)

Fig. 8b

Ta-40W
\( \sigma_{f0}^* = 971 \text{ MPa, } \sigma_{w0} = 750 \text{ MPa} \)
DBTT_0 = 153 K at \( d_k = 25 \mu m \)
\( \varepsilon_{f0} = 0.4, 1.8 \leq k_i \leq 2.6 \)
Fig. 9

ODS-Ta-40W

$\sigma_{f,o}^* = 1200 \text{ MPa}, \ \sigma_{y_0} = 750 \text{ MPa}$

$f_v = 0.001, \ d_p = 12 \text{ nm}$

$\varepsilon_{f,o} = 0.4, \ 1.8 \leq k_i \leq 2.6$

$V_o = \frac{\Delta DBT_{tot}}{USE_o}$

$U_p = \frac{\Delta USE_{dk}}{USE_p}$

$\text{grain refinement (nm)}$
Fig. 10

--- $\sigma_f=\text{const.}$ below $d_k=500$ nm

maximum

minimum

$\sigma_{f_0}=1440$ MPa, $\sigma_c=441.7$ MPa
$\varepsilon_{f_0}=0.4$, $1.8 \leq \kappa \leq 2.6$, $\sigma_y=725$ MPa

$\leftarrow$ experimental

$\leftarrow$ model
$c_f=5$

--- $d_k$ (nm)
<table>
<thead>
<tr>
<th>Metal</th>
<th>b (nm)</th>
<th>μ (GPa)</th>
<th>Tm(°C)</th>
<th>Λth (W cm⁻¹K⁻¹)</th>
<th>αth (10⁻⁶)</th>
<th>Θ</th>
<th>decay time of main isotopes</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-Fe</td>
<td>0.2482</td>
<td>71.8</td>
<td>1536</td>
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<td>2996</td>
<td>0.575</td>
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<td>2610</td>
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<td>3</td>
<td>66 h</td>
</tr>
<tr>
<td>Cr</td>
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<td>3152</td>
<td>1.037</td>
<td>5.58</td>
<td>5.5</td>
<td>98 d</td>
</tr>
</tbody>
</table>

Tab. 1: Main physical properties of Ta, W, Ta-40at%W and Mo, Cr which compared with α-Fe. (d..days, h...hours, Θ..cross section for 14MeV neutrons)