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Microstructure evolution and creep strength of new-generation oxide dispersion strengthened alloys with high volume fraction of nano-oxides

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Abstract

Four creep resistant Fe-based oxide dispersion strengthened (ODS) alloys with a significantly high amount of dispersed oxide nanoprecipitates have been investigated. One Fe-Al-O and three Fe-Al-Cr-Mo-Y_2O_3 systems with different chemical composition strengthened by yttrium nano-oxides have been prepared by mechanical alloying of powders and consolidation by hot rolling leading to ultrafine grained microstructure due to dynamic recrystallization. The thermal stability of the precipitates, effects of the processing on the microstructure (grain and precipitate size) and mechanical properties at high temperatures have been evaluated. It has been found that the rolling temperature has a significant effect on the static recrystallization process during the subsequent heat treatment and on the resulting grain size of the alloys and does not affect the size of nano-oxides and their dispersion. Tensile tests performed at a low constant rate of 10^{-6} s^{-1} allow a quick estimate of the creep strength and helps to a quick identification of optimum processing conditions. It was found that the mechanism of the fracture is changing from trans-granular to inter-granular between 600 and 800 °C which leads to a significant drop of ductility. The set of mechanical tests has been completed also for 1100 °C indicating a rather high applicability potential of the investigated system.

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Keywords: nano-oxide; dispersion; creep; fracture; high temperature

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1. Introduction

The oxide dispersion strengthened (ODS) alloys represent a group of materials for high temperature applications, such as in 4-th generation of fission or fusion reactors, aero jet turbine blades, high temperature grips for testing machines etc. Their microstructure consists of the steel matrix strengthened by dispersion of stable yttrium-based oxides of typical size between 5-30 nm and of volume fraction of about 0.5 %. A processing route of the ODS steels involves two steps: (i) the nano-composite powder containing matrix and yttria nanoparticles is produced by mechanical alloying (MA) and (ii) the powder is then hot consolidated. Several commercial alloys have been developed such as MA956 or MA957, PM 2000 or PM2010, ODM alloys and 1DK or 1DS. The non-commercial, experimental and advanced versions of ODS alloys are ODS Eurofer, 9YWT, 12YWT and 14YWT. Excellent creep strength of the ODS steels is associated with an attractive interaction between dislocations and oxides. The main disadvantage is the low creep fracture strain which represents a limiting factor for use in practice. The aim of the ongoing effort is to explore the Fe-Al-O and Fe-Al-Cr-Mo-Y-O systems with high oxygen content (up to 1.5 wt. %) represented by alumina or yttria oxides (up to 5 vol. %) to identify the potentials of new generation of ODS alloys. It has been studied at high strain rates by Mašek et al. (2016) with promising results. A kinetic study of Fe-Al-O alloy recrystallization was performed by Bártková et al. (2017). The influence of thermomechanical treatment on the grain-growth behaviour of new Fe-Al based alloys with fine Al_2O_3 precipitates was studied by Khalaj et al. (2017). The thermal stability and coarsening of the dispersed oxides with Al_2O_3 and Y_2O_3 was studied by Svoboda et al. (2018). It was shown that the stability of Y_2O_3 precipitates is much better than Al_2O_3 at temperatures over 1000 °C. The mechanical properties of the new ODS alloys up to 800 °C were studied by Dymáček et al. (2019). The aim of this paper is to compare alloys of different chemical composition and the effect of the rolling temperature on mechanical properties up to 1100 °C.

2. Materials and methods

The manufacturing process of the studied alloys is still in the optimization phase. In this case one Fe-Al-O (A1) and three Fe-Al-Cr-Mo-Y_2O_3 (M1, M2, M9) systems with different chemical composition (see Tables 1 and 2) strengthened by yttrium nano-oxides have been prepared. The process consists of powder preparation by mechanical alloying in a ball mill (own design) in controlled oxygen atmosphere (A1) or in vacuum (M1, M2, M9), during which the oxygen is captured at manifold microstructural defects such as dislocations and vacancies in the heavily deformed powder, see Bártková et al. (2017). The small amount of Ni and Co is due to use of maraging steel balls in the mill in order to lower the carbon content and to prevent formation of carbides in the alloys. After consolidation of the powder by hot rolling in a steel container during three rolling steps at different temperatures ranging from 750 °C to 1060 °C, an ultra-fine-grained (UFG) microstructure is obtained due to dynamic recrystallization, see Fig. 1a. The third rolling temperature has been varied to see the effect on the static recrystallization during subsequent heat treatment and mechanical properties afterwards. The manufacture of tensile samples was performed by water jet cutting, the samples were with squared section 3 x 3.2 mm, and gauge length 25 mm. The heat treatment and static recrystallization was performed on already manufactured samples on air. During this heat treatment protective Al_2O_3 layer is formed. The alloy A1 was annealed at 1100 °C 4h, the M1, M2 and M9 alloys were annealed at 1200 °C 16h. The microstructure after static recrystallization is shown in Fig. 1b and the fine dispersion of nano-oxides in Fig. 1c.

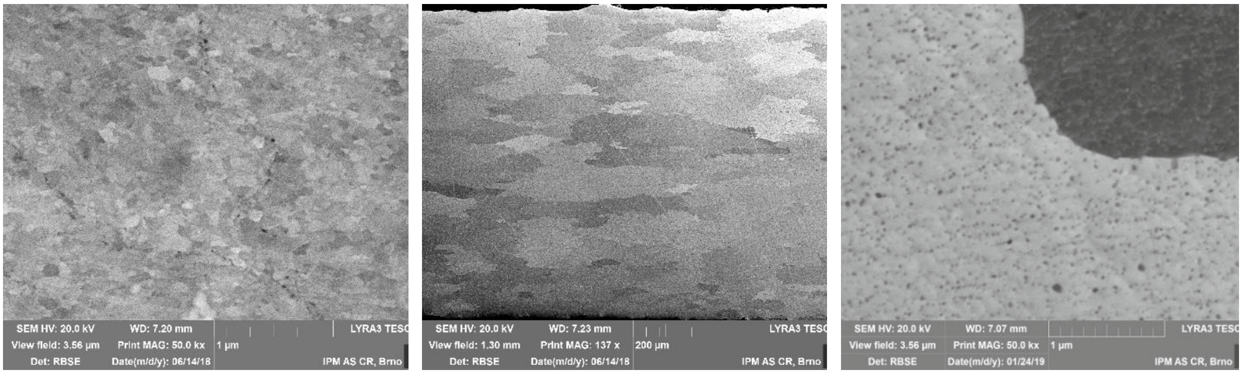
The scanning electron microscope Tescan Lyra 3 XMU was used for a study of the specimen microstructure and oxide dispersion as well as specimen fracture surfaces. Creep testing machine Messphysik KAPPA LA 50 kN equipped with Maytec vacuum furnace up to 1400 °C was used for the low strain rate tensile experiments at different temperatures.

Table 1. Chemical composition of A1 alloy in wt. %.

Alloy	Fe	Al	O	Ni	Co
A1	86.4	11	1.1	0.9	0.6

Table 2. Chemical composition of M1, M2 and M9 alloys in wt. %.

Alloy	Fe	Al	Cr	Y_2O_3	Mo	Ni	Co
M1	71.8	6.2	13.7	3.4	3.4	0.9	0.6
M2	73.6	3.6	14.1	3.6	3.6	0.9	0.6
M9	68.9	9.2	13.2	3.6	3.6	0.9	0.6



(a) (b) (c)

Fig. 1. M1 alloy (a) after consolidation by hot rolling, (b) coarse grained microstructure and (c) dispersion of nano-oxides in adjacent coarse grains after static recrystallization.

3. Results and discussion

Prior the time consuming creep testing a tensile testing at slower constant rate (10^{-6} s^{-1}) is beneficial for the optimization process of the developed alloys. Tensile curves of M1 alloy at different temperatures are shown in Fig. 2. It is obvious that the ductility dropped significantly between 600 and 800 °C and it is caused by changing mechanism of the fracture. While it is mainly transgranular ductile fracture with characteristic dimples up to 600 °C, there is more presence of the fracture along the grain boundaries and cracks between the elongated grains at 800 °C and above.

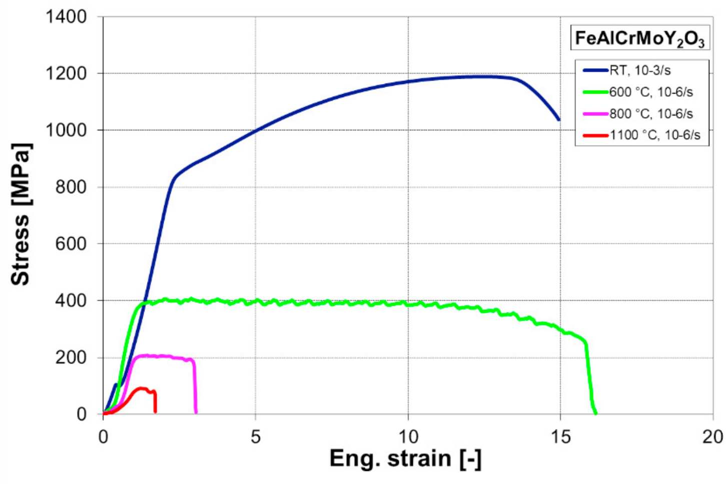
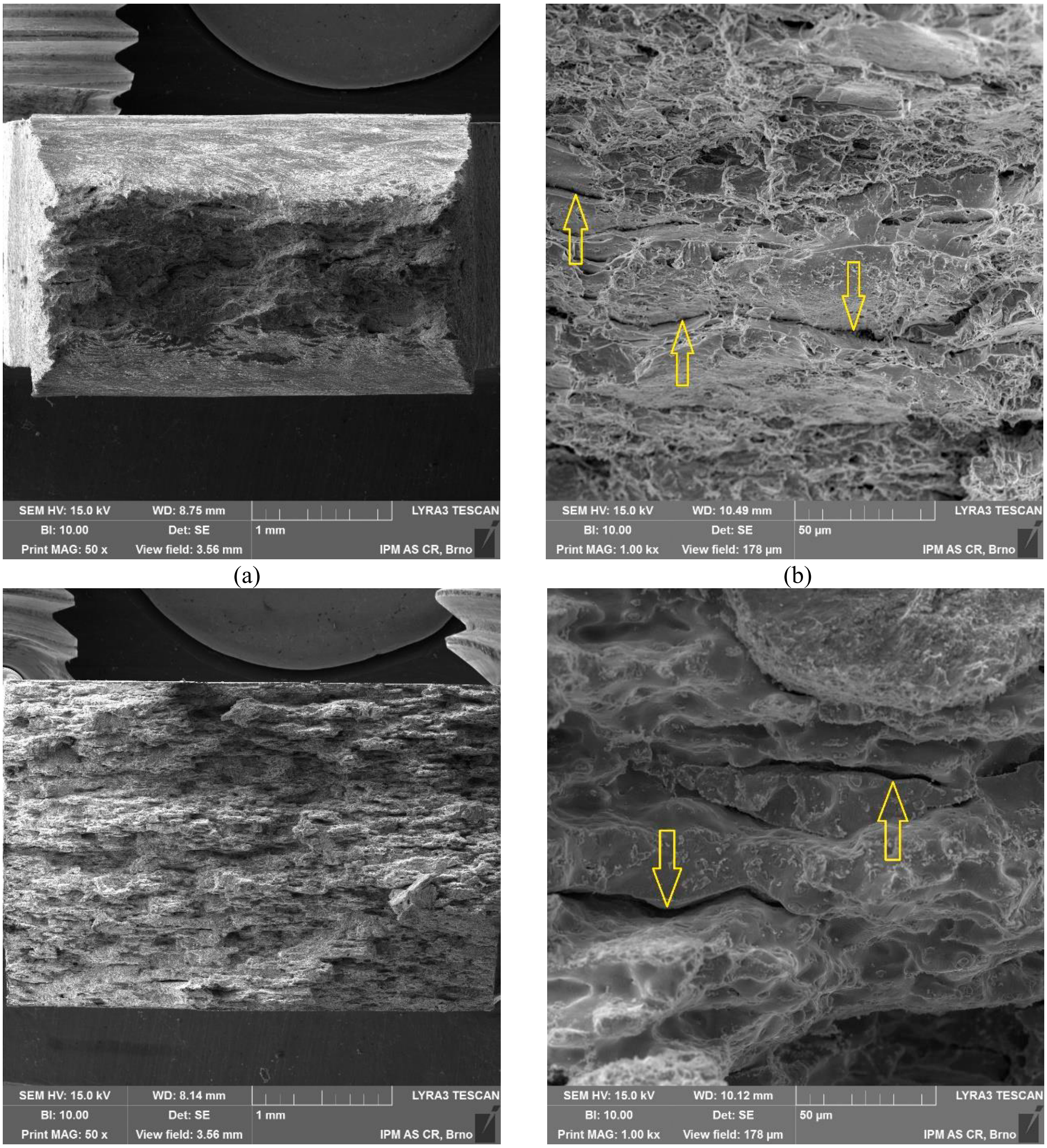


Fig. 2. Illustration of M1 alloy tensile curves at various temperatures.

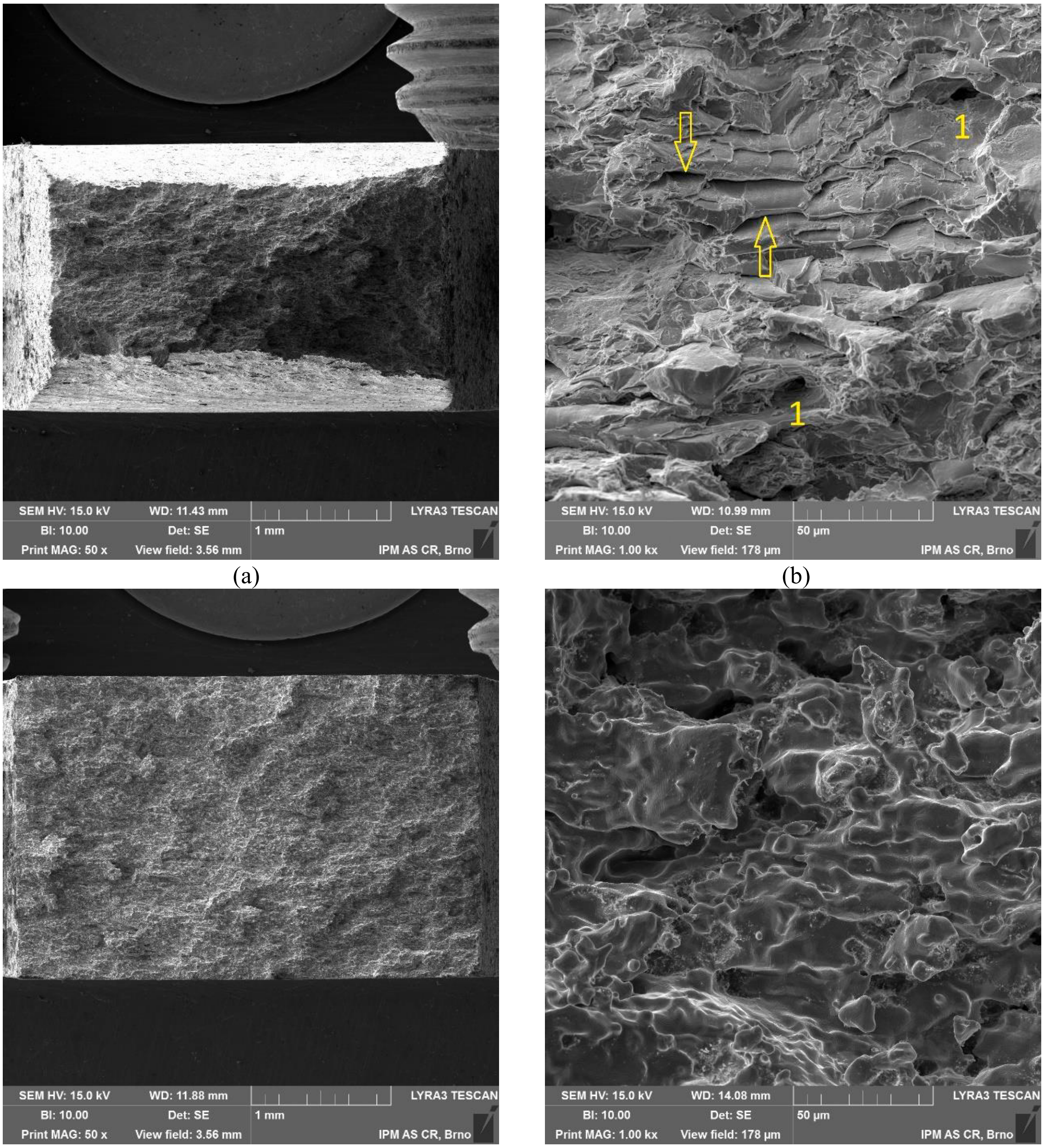
The fracture surfaces of the M9 alloy at 600 and 1100 °C are documented in Fig. 3. It is possible to distinguish the necking at 600 °C in Fig. 3a compared to practically un-necked fracture surface at 1100 °C in Fig. 3c. The ductile fracture morphology with characteristic dimples is present in Fig. 3b, however some signs of cracks between flat grains are marked by arrows. These intergranular cracks are more visible at 1100 °C in Fig. 3d.



(a) (b)

(c) (d)

Fig. 3. Fracture surfaces of M9 alloy after tensile test at 600 °C, 10^{-6} s^{-1} (a) 50x, (b) 1000x magnification, and 1100 °C, 10^{-6} s^{-1} (c) 50x, (d) 1000x

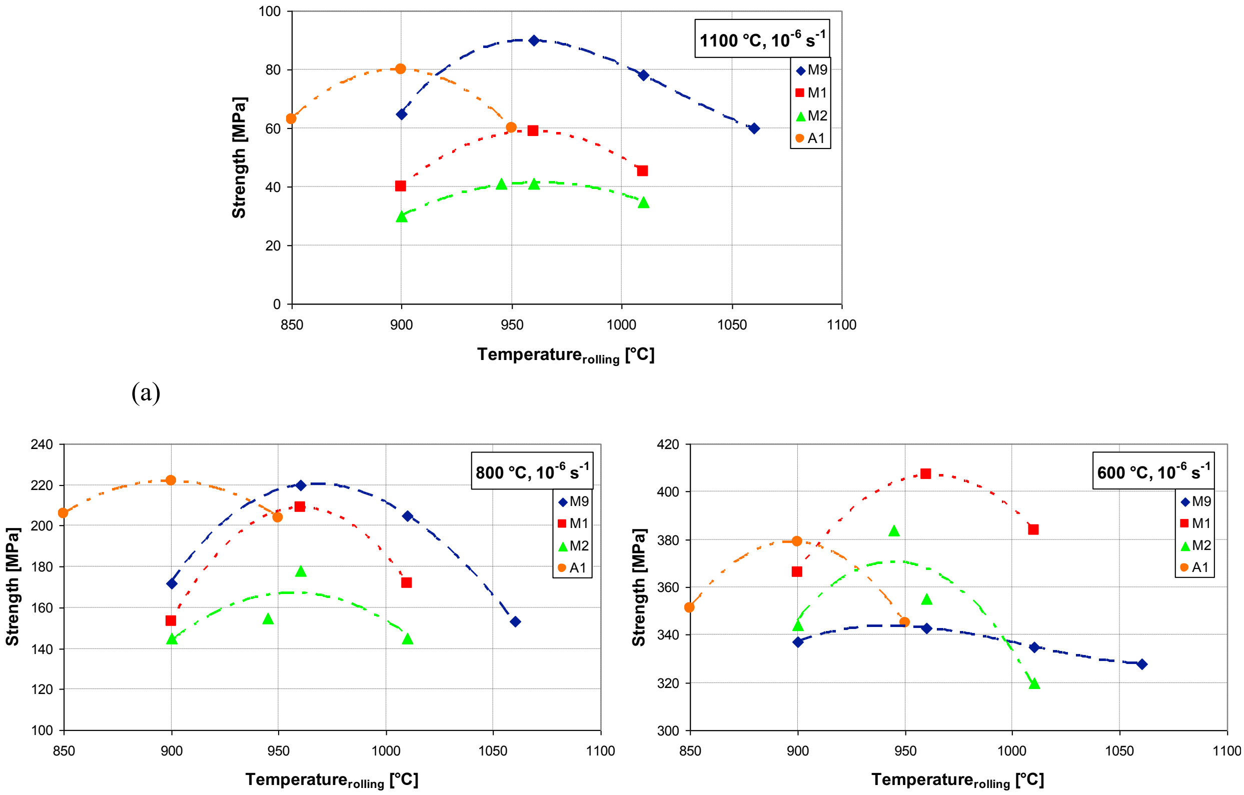


(a) (b)

(c) (d)

Fig. 4. Fracture surfaces of A1 alloy after tensile test at 600 °C, 10^{-6} s^{-1} (a) 50x, (b) 1000x magnification, and 1100 °C, 10^{-6} s^{-1} (c) 50x, (d) 1000x

The effect of rolling temperature was studied for all four alloys as shown in Fig. 5. The best alloy at 1100 °C is M9 with the highest Al content. At the lower temperatures dominate the A1 and M1 alloys. Considering all test temperatures, the third rolling temperature between 960°C seems optimal for M1-M9 alloys and 900°C for A1 alloy.



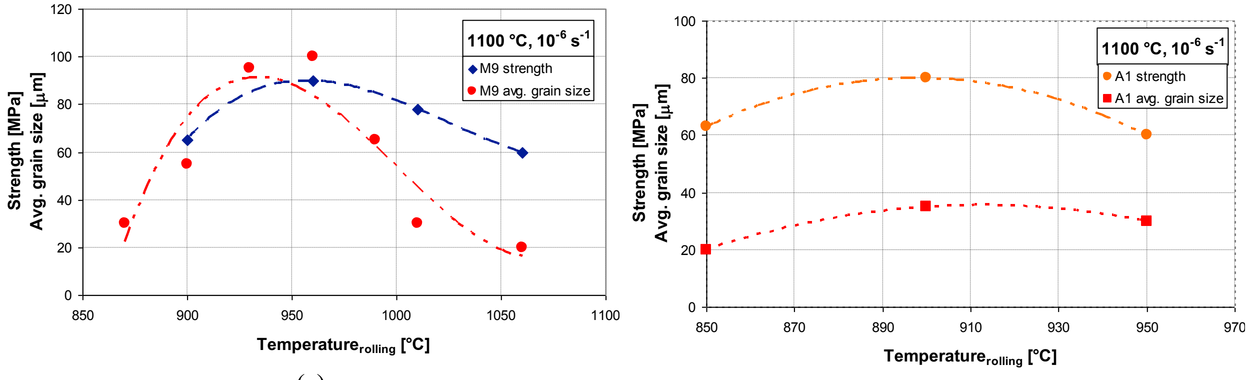
(a)

(b)

(c)

Fig. 5. Strength of studied alloys depending on the rolling temperature (a) 1100 °C, (b) 800 °C, (c) 600 °C.

The average grain size dependence on the rolling temperature was correlated with strength at 1100 °C for M9 and A1 alloys in Fig. 6. The highest strength occurs in the case of largest grain size. This is in agreement with the creep behavior of polycrystalline materials since the grain boundaries act as an accelerator of creep deformation and damage.

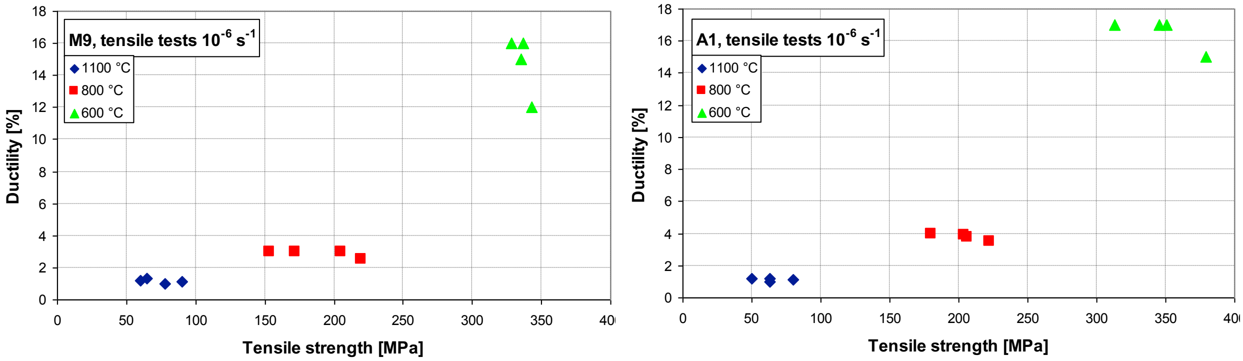


(a)

(b)

Fig. 6. Strength and average grain size of (a) M9 and (b) A1 alloys depending on the rolling temperature.

The strength-ductility diagrams shown in Fig. 7 for M9 and A1 alloys show dramatic drop of ductility between 600 and 800 °C and also shows the differences in strength due to different rolling temperatures.



(a) (b)

Fig. 7. Strength-ductility diagram of (a) M9 alloy (b) A1 alloy.

4. Conclusions

Presented ODS alloys contain about one order of magnitude more nano-oxides in dispersion than the classical ODS alloys. The ductility is high between RT and 600 °C. Above 800 °C there is notable decrease of ductility presumably due to lower cohesion strength of the grain boundaries. The temperature of the last rolling step has a significant influence on the grain size after static recrystallization and therefore also on creep strength.

The rolling temperature of 960 °C was found as optimal for the high temperature strength of M1, M2 and M9 alloys. The rolling temperature of 900 °C is optimal for A1 alloy. The M9 seems to be as the most promising alloy for applications above 1000 °C while the A1 alloy suits for applications up to 1000 °C.

The grain boundaries weakening above 800 °C is still under intensive investigation. Further optimization of new generation of ODS alloys is still ongoing.

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