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Plasticity of oxide dispersion strengthened ferritic alloys

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ABSTRACT

Two 13%Cr oxide dispersion strengthened (ODS) ferritic alloys, DT and DY, exhibiting different oxide particle size distribution and a χ phase precipitation were studied. Their tensile properties have been tested from 20 to 700°C. Experimental observations during room temperature tensile tests performed in a scanning electronic microscope have shown that the main damage mechanism consists in microcracking of the χ phase precipitates on grain boundaries. These alloys are high tensile and creep resistant between 500 and 700°C. Their strongly stress-sensitive creep behaviour can be described by usual creep laws and incorporating a threshold stress below which the creep rate is negligible.

INTRODUCTION

Ferritic alloys irradiated as core components of a fast neutron reactor in the range from 400 to 650°C are more resistant to swelling than austenitic ones (1). In order to improve their creep properties, ferritic alloys have been strengthened by oxide dispersion. The ODS alloys are elaborated by mechanical alloying, a process derived from powder metallurgy, compaction and extrusion (2). Up to now the mechanical behaviour of Fe or Ni base ODS alloys is not very well understood (3). The high temperature behaviour of ODS alloys during creep tests is unusual : the stress sensitivity of the strain rate is very high. Values of stress exponent, usually near 3-5 in single phase materials, are above 10 (4, 5). To describe this particular behaviour the semi-empirical equations involve a threshold stress below which the strain rate becomes negligible. At low temperatures, the threshold stress may be compared with the Orowan stress due to particle dispersion (6). Nevertheless at high temperatures, the deformation mechanisms are still not well understood. Arzt et al. have proposed a theoretical model of dislocation processes based on dislocation detachment from dispersoid particles (4).

The main goal of the present work is to get a better understanding of the viscoplastic behaviour of ODS ferritic alloys in order to improve their mechanical properties. In this paper, tensile tests results of two ODS ferritic alloys exhibiting an intermetallic χ phase are given as a function of temperature and axial creep data in the range from 500 to 700°C are presented and discussed.

EXPERIMENTAL PROCEDURES

Materials

Two ODS ferritic alloys, labelled DT (DT2906) and DY (DT2203Y03), developed by CEN/SCK of Belgium and elaborated by DOUR Metal (7), have been studied. They were prepared by mechanical alloying, followed by extrusion sintering, a 15 minute treatment at 1050°C and a final one day treatment at 800°C. The chemical analysis of DT and DY alloys is reported in Table 1. Both alloys exhibit an abundant

grain boundary precipitation of a stable bcc FeCrTiMo intermetallic χ phase during the 800°C thermal treatment (8). DT contains Ti_2O_3 dispersions whereas DY is strengthened by Ti_2O_3 and Y_2O_3 . They are quite different from the oxide distribution point of view as discussed further on.

Table 1 : Chemical composition of ODS ferritic alloys (wt%)

Elements (wt.%)	Cr	Mo	Ti	O2 bound to Ti	Y ₂ O ₃
DT (DT2906)	13	1,5	2,9	0,6	—
DY (DT2203Y05)	13	1,5	2,2	0,3	0,5

Microscopic observations

Optical metallography is carried out on specimens etched in gliceregia's reagent.

Plate tensile specimens are mechanically polished and etched in gliceregia's reagent, and examined in a JEOL JSM 35C scanning electronic microscope (SEM).

Thin foils suitable for transmission electron microscopy (TEM) are prepared by jet electropolishing in a Struers Tenupol unit at 30V and 13°C, using an electrolyte of 10% perchloric acid in a mixture of ethyl alcohol with 20% ethylenglycol-monobutylether. The foils are examined in a Philips EM430 electron microscope operating at 300kV.

Tensile and creep tests

The DT and DY alloys are supplied in the form of extruded bar of diameter 9 mm. Tensile and creep specimens of 4 mm in diameter with a 20 mm gauge length, and plate tensile specimens of 0,5 x 3 mm² cross section with 20 mm gauge length are machined with their longitudinal axes parallel to the extrusion direction. Constant-load creep tests are carried out over a range of stresses at temperatures varying from 500 to 700°C. The strain tensile rate is 8.10⁻⁴s⁻¹. Broken specimens are examined after tensile or creep test in a JEOL JSM 35C SEM.

EXPERIMENTAL RESULTS

Microstructure

Typical micrographies from a longitudinal section of DT and DY bars are presented in figure 1, and illustrate a grain structure elongated in the direction of extrusion with χ phase precipitation on grain boundaries.

Figure 1 : Optical micrographies illustrating the elongated grain structure and χ phase precipitation on grain boundaries in DT and DY alloys (longitudinal section).

Oxide dispersion

Based on TEM observation of thin foils, and image analysis, the measured diameters of the nearly spherical particles are concentrated around 10 and 30 nm in DY whereas the size distribution of DT is much larger and ranges from 40 to 120 nm (figures 2 and 3). Moreover, the mean diameter of the particles in DT is almost four times greater than in DY.

Figure 2 : Particle size distribution in DT alloy

Figure 3 : Particle size distribution in DY alloy

Tensile properties

Tensile properties have been measured in the longitudinal bar direction for test temperatures in the range from 20 to 700°C. Yield and ultimate tensile stresses and elongation of DT and DY as a function of temperature are shown respectively in figures 4 and 5. A minimum in elongation is observed at about 400°C, followed by a pronounced improvement in high temperature ductility. The decrease of the tensile strength of these alloys is more pronounced from 500 to 700°C. Moreover, the lower strength of DT, associated to a better ductility, appears clearly, as compared with DY mechanical properties.

Figure 4 : Variation of yield and ultimate tensile stresses (YS and UTS) with temperature for DT and DY alloys

Figure 5 : Variation of total tensile elongation with temperature for DT and DY alloys

Examination of fracture surfaces and fracture profiles reveals that failure occurs by nucleation, growth and coalescence of microcracks initiated in the χ phase.

Continuous observations during tensile test performed at room temperature inside the chamber of a scanning electronic microscope show that the occurrence of microcracking of the χ phase coincides with macroscopic yielding and initiates the ductile tearing of the matrix. The microstructure of the fracture profile of DT just before failure in figure 6 clearly illustrates these two consecutive sequences of the fracture process.

Figure 6 : (a) Microcracking of the χ phase and (b) ductile tearing of the matrix during room temperature tensile test on DT alloy (SEM observation).

Creep properties

Creep tests were performed in air at 500, 650 and 700°C. Figure 7 summarizes the creep characteristics of DT and DY tested in the longitudinal direction. These data reveal that for rupture life ranging from 1h to more than 1000h and for temperature between 500 and 700°C, a stress about 100 MPa higher than for DT alloy can be applied to DY alloy.

Figure 7 : Influence of stress on rupture life of DT and DY alloys as a function of temperature

DISCUSSION

Tensile properties

DT and DY alloys present different tensile behaviour as a function of temperature. DY is more resistant but less ductile than DT. It must be noted that the originality of DT and DY consists in the χ phase precipitation at grain boundaries, which does not exist in other ODS ferritic alloys, as MA956 and MA957 (9). It seems that, according to the observations on the tensile test in the SEM, the χ phase acts as a brittle defect in a ductile matrix. Now compared with the tensile properties of MA956 and MA957, DY is as strong as these materials and less ductile whereas DT shows a similar ductility. According to Orowan's mechanism, better elevated temperature mechanical properties can be achieved by both fine dispersoid sizes and fine interparticle spacing. So, owing to the finer dispersoid size in DY (fig.3) and assuming that the interparticle spacing is finer for DY, we can attribute the higher strength of DY to its finer oxide dispersion. A quantitative analysis of the interparticle spacing has been presently undertaken. The contribution of the χ phase should be less important.

Creep properties

The stress and temperature dependance of the creep rate can be described by the classical semi-empirical equation :

$$\dot{\epsilon} = A D (\sigma / E)^n$$

where A is a material parameter, n is the stress exponent, D is the diffusion coefficient and E is the elastic modulus at various test temperatures (3). Figure 9 shows a plot of the normalized strain rate $\dot{\epsilon} / D$ as a function of the normalized stress σ / E for DT.

Figure 9 : Stress dependance of the normalized creep rate $\dot{\epsilon} / D$ for DT

It is obvious that this equation well describes the deformation behaviour of DT, but the stress exponent n, usually ranging from 3 to 5, is greater than 10. DY exhibits a similar behaviour. Such a high stress sensitivity is described by invoking a threshold stress σ_{th} below which the creep rate is considered to be negligible. It can be rationalized as :

$$\dot{\epsilon} = A D \left(\frac{\sigma - \sigma_{th}}{E} \right)^{n'} \text{ with } n' \text{ ranging from 3 to 5.}$$

In many models, σ_{th} turns out to be some fraction of Orowan stress (10, 11). Its physical meaning is still controversial (12-16). One of the theories suggested in explaining this phenomenon is the dislocation bypass over the dispersoid through local climb, as proposed by Arzt et al. (17, 18). The strongest barrier to dislocation bypass is no longer provided by the climb but rather by the detachment of the dislocation from

bypass over the dispersoid through local climb, as proposed by Arzt et al. (17, 18). The strongest barrier to dislocation bypass is no longer provided by the climb but rather by the detachment of the dislocation from the particle after climb over the particle is completed, due to an attractive force exerted by particles on the dislocations.

Assuming that 10^{-9}s^{-1} is a negligible creep rate, the threshold stress for DT is estimated to be about 225MPa at 500°C, 90MPa at 650°C and 65MPa at 700°C. It is estimated to be about 340MPa at 500°C, 180MPa at 650°C and 157MPa at 700°C for DY. But these estimates should be viewed as preliminary. Dip tests are under way to better determine this threshold stress (19).

CONCLUSION

- 1) Tensile tests performed from 20 to 700°C, and creep tests between 500 and 700°C of two 13%Cr ODS ferritic alloys, have shown that the DY alloy is stronger and less ductile than the DT alloy. It is consistent with the finer yttrium oxide dispersion of DY.
- 2) Tensile failure at room and elevated temperatures in DT and DY occurs by microcracks of the χ phase precipitated on grain boundaries followed by the ductile failure of the matrix. Nevertheless mechanical strength, compared with ODS ferritic alloys without χ phase as MA956 and MA957, is mainly determined by the oxide dispersion in the matrix rather than by the χ phase.
- 3) As many ODS alloys, the creep behaviour of DT and DY is characterized by a high stress sensitivity of the deformation. It can be compensated by introducing a threshold stress below which the creep rate becomes very low.

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