MECHANICAL PROPERTIES OF ULTRA-FINE GRAINED ODS Fe-Cr BASED ALLOYS

M. A. Auger, V. de Castro,T. Leguey, A. Muñoz, M.A. Monge, R. Pareja
Departamento de Física. Universidad Carlos III de Madrid. 28911 Leganés, Spain
mauger@fis.uc3m.es

ABSTRACT

In the present work, ODS and non-ODS alloys with target compositions: Fe-14Cr, Fe-14Cr-0.3Y_2O_3 and Fe-14Cr-2W-0.3Ti-0.3Y_2O_3 (% wt) have been produced by mechanical alloying, canning and degassing, HIP, forging and heat treatment. The milled powder was analysed by scanning electron microscopy (SEM), laser diffraction (LD) and the O and C contents were quantified. The microstructure of the obtained billets has been investigated by X-ray diffraction (XRD) and transmission electron microscopy (TEM) techniques. The density and the Vickers microhardness have been measured and tensile tests in the temperature range 273-973 K have been performed for all the alloys. A homogeneous dispersion of oxide nanoparticles was observed in the ODS alloys, and the tensile properties were enhanced in comparison to the Y_2O_3 free alloy. The obtained results demonstrate that the powder metallurgy route applied in the present work can produce ODS ferritic Fe-Cr alloys with enhanced microstructure and mechanical properties.

INTRODUCTION

Irradiation resistance, high-temperature strength and reduced activation are the main properties demanded for the structural materials to be used in the future nuclear reactors in order to build devices with improved efficiency and safety. Among the candidate materials, oxide dispersion strengthened (ODS) reduced activation ferritic (RAF) steels appear to be the most promising candidates with such properties. The increasing interest in developing ODS RAF steels relies on the experimental results, which have shown improved irradiation resistance, high-temperature properties and thermal stability. The microstructure and composition of ODS materials, which depend on how they are processed, are the key for achieving the desired properties. The aim of the present work is the development of an ODS RAF Fe-Cr model alloy with an enhanced ultra-fine grained microstructure and the assessment of its mechanical properties.

EXPERIMENTAL

Mechanical alloy of elemental powders

Milling parameters:

- Chromium steel vessels and balls.
- Balls to powder ratio 10:1.
- Rotation speed: 300 rpm.

Canning of alloyed powders

- 304L stainless steel cans
- Vacuum degassing at 823 K for 24 h.

HIP of the canned powders

- 1373 K and 200 MPa for 2 h

Forging

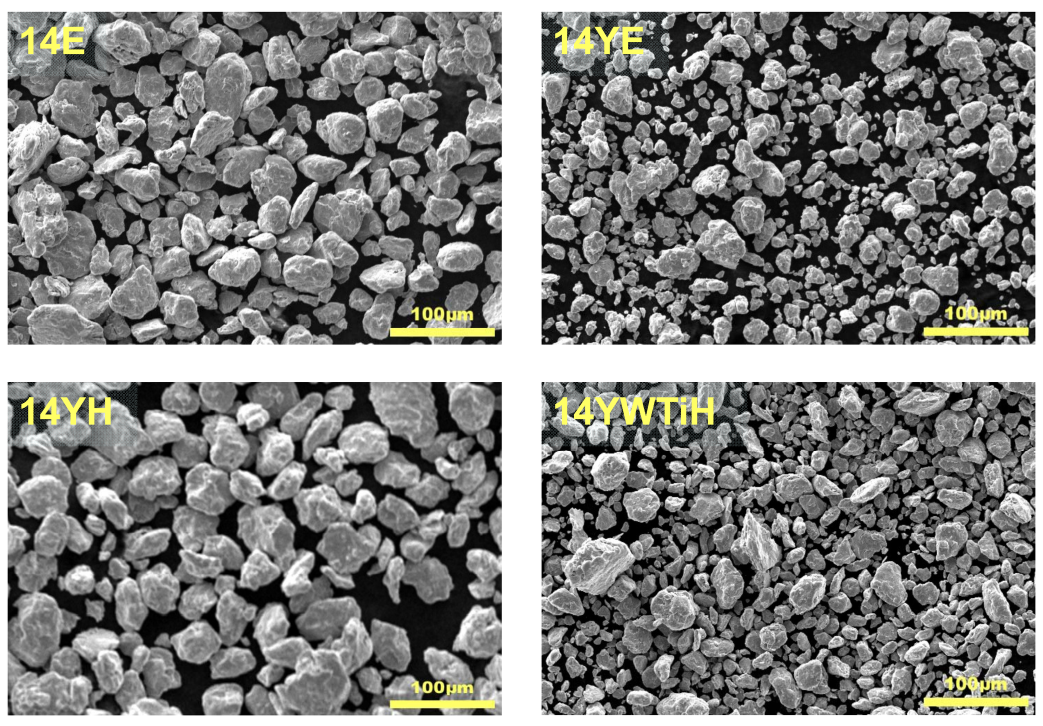
- 14E at ~1323 K
- 14YE and 14YH at ~1373 K
- 14YWTiH at ~1423 K

Thermal treatment

- 14E at 1123 K for 1 h, air cooled
- 14YE, 14YH, 14YWTiH at 1123 K for 2 h, air cooled

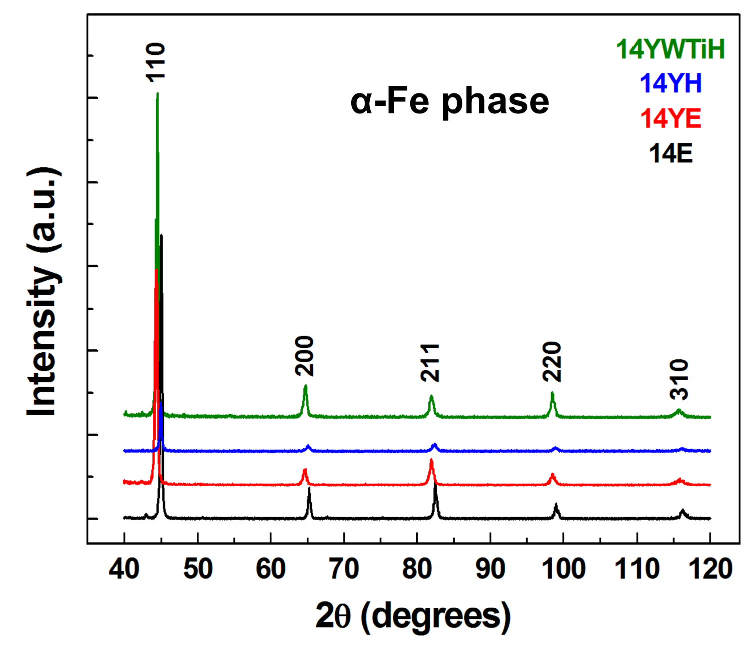
Sample designation	Nominal composition (wt %)	Milling atmosphere	Milling time	Milling device
14YWTiH	Fe-14Cr-2W-0.3Ti-0.3Y_2O_3	H	48 h	Retsch PM400
14YH	Fe-14Cr-0.3Y_2O_3	H	60 h	Fritsch Pulverisette 6
14YE	Fe-14Cr-0.3Y_2O_3	He	60 h	
14E	Fe-14Cr	He	60 h	

POWDER CHARACTERIZATION



Sample	O (wt%)	C (wt%)	Particle size (µm)
14YWTiH	0.781	0.059	22.91
14YH	0.427	0.063	31.70
14YE	0.432	0.095	35.56
14E	0.406	0.057	44.77
Fe powder	0.28	0.022 – 0.050	< 10 µm
Cr powder	0.55	0.005 – 0.007	< 10 µm
W powder	0.2245	-----	APS 1-5 µm
Ti powder	0.068	0.013 - 0.050	150 mesh (~ 100 µm)
Y_2O_3 powder	-----	-----	15 – 30 nm

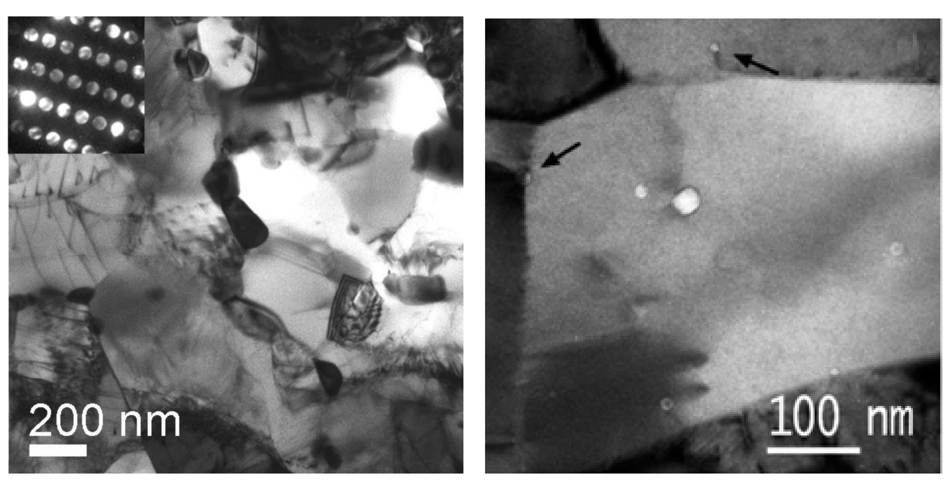
XRD, DENSITY AND MICROHARDNESS



Sample	Lattice parameter (Å)	Density (g/cm³)	Microhardness (GPa)
14YWTiH	2.879 ± 0.001	7.728 ± 0.018	3.70 ± 0.06
14YH	2.865 ± 0.008	7.481 ± 0.176	3.65 ± 0.08
14YE	2.878 ± 0.004	7.721 ± 0.022	4.16 ± 0.05
14E	2.858 ± 0.009	7.642 ± 0.014	2.70 ± 0.04

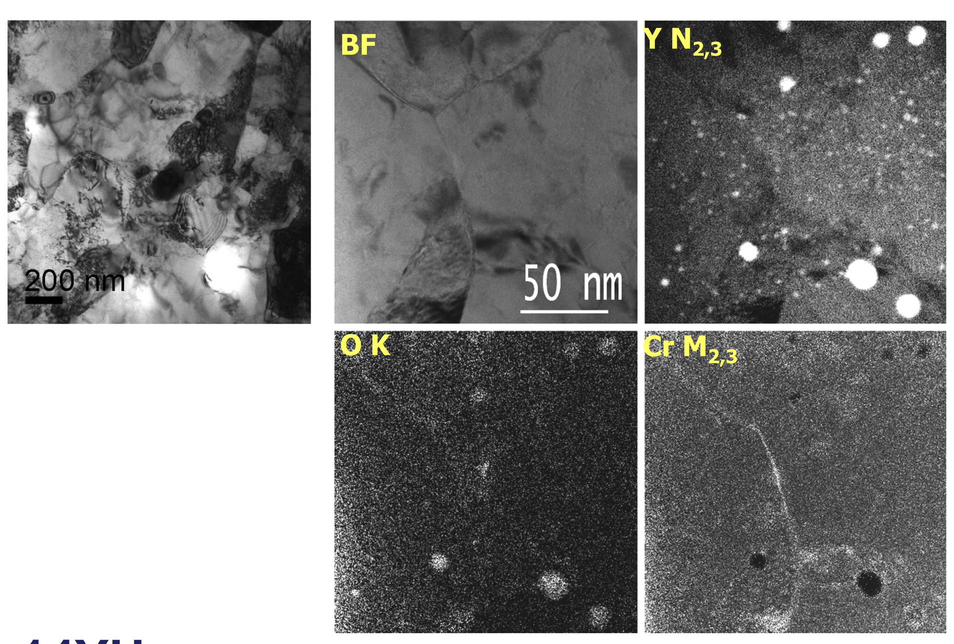
MICROSTRUCTURE

14E



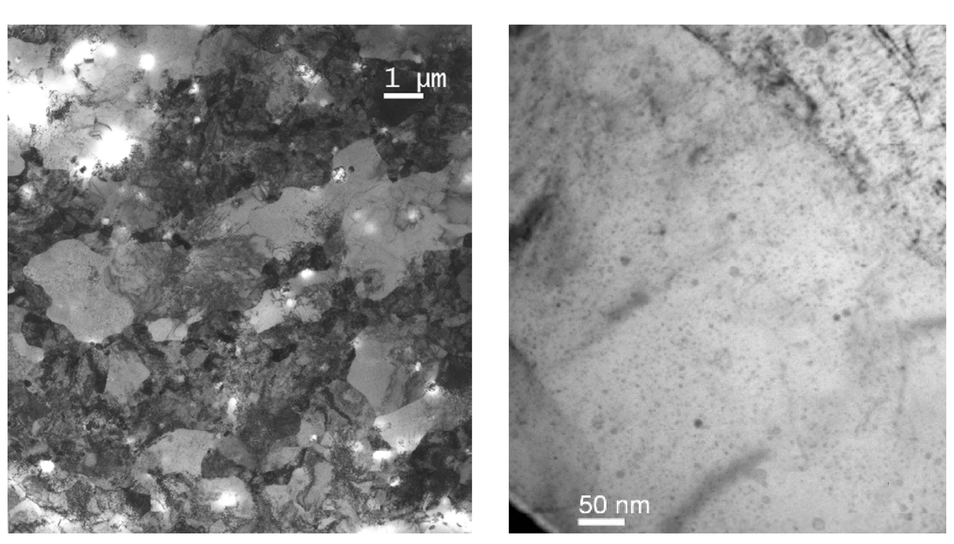
- Duplex microstructure: micron and submicron sized grains
- Cr rich precipitates at grain boundaries and within grains with sizes ~ 50 – 400 nm. Indexed as Cr_2O_3 or $M_{23}C_6$ (M=Cr, Fe)
- Voids with sizes ~ 5 – 25 nm found within grains and attached to dislocations, grain boundaries and Cr-rich precipitates

14YE



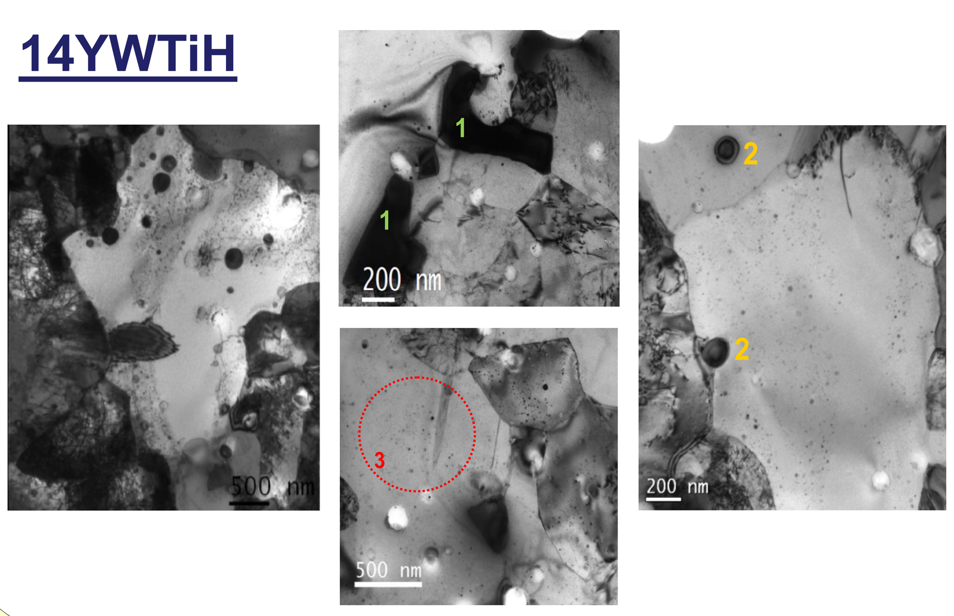
- Only submicron sized grains
- Nanoparticles distributed mostly within grains, also pinning grain boundaries and dislocations
  Sizes ~ 1 – 30 nm, most of them < 10 nm
  Density of particles: between 0.10 ± 0.02 and 1.5 ± 0.3 (x10^{23} m^{-3})
- All the studied nanoparticles were Y-rich. Most particles had a core-shell structure consisting of an Y-O rich core and a Cr-rich shell
- Voids with sizes < 5 nm within grains, also frequently attached to Y-rich particles and dislocations, grain boundaries and Cr-rich precipitates

14YH



- Uniform grain structure: 600 – 1000 nm sized grains
- Uniform distribution of Cr-rich precipitates
- No voids observed
- Density of particles: 1.2 x10^{21} m^{-3}

14YWTiH



- Duplex microstructure: - dislocation-free grains up to 15 µm in length
  - equiaxed submicron grains sized ~ 400 – 800 nm
- Different types of particles found:
  - Type 1→ Cr-W rich, sizes up to ~ 1.5 µm. Indexed as $M_{23}C_6$ (M=Cr, W)
  - Type 2→ Cr-Ti rich, sizes up to ~ 100 – 200 nm
  - Type 3→ Y-Ti rich, sizes < 30 nm, most of them < 10 nm. Indexed as Y_2TiO_5
    Density of particles: between 3.7 ± 0.7 and 13 ± 3 (x10^{21} m^{-3})

MECHANICAL PROPERTIES