2.1 Fe-Cr-Al ODS ALLOYS FOR FUSION REACTOR APPLICATIONS—S. Dryepondt , K. A. Unocic, D. T. Hoelzer (Oak Ridge National Laboratory)

OBJECTIVE

The dual coolant lead-lithium (DCLL) blanket concept requires ferritic steels with improved Pb-Li compatibility in order to demonstrate viable blanket operation in a DEMO-type fusion reactor. This project aims at developing alumina –forming oxide dispersion strengthened (ODS) FeCrAl alloy with excellent compatibility with Pb-Li and creep resistance at temperature up to 700-800ºC.

SUMMARY

Several ODS FeCrAl alloys were fabricated by ball milling Fe-12Cr-5Al gas atomized powder with Y_2O_3 powder and ZrO_2, HfO_2, or TiO_2 powder. The Zr-containing alloy (125YZ) exhibited excellent creep properties at 700-800ºC in air, and good compatibility with Pb-17Li at 700ºC, with the formation of a thin $LiAlO_2$ layer. Extensive TEM characterization of the 125YZ revealed the presence of $Y_3Al_5O_{12}$ (YAG), Al_2O_3 and Zr(C,N) nano precipitates. A second generation of ODS FeCrAl alloys was fabricated by ball-milling Zr or Hf-containing Fe-12Cr-5.5Al powder with Y_2O_3 powder. Initial characterization of these 2nd generation alloys showed that the alloy tensile properties are very dependent on the extrusion conditions.

PROGRESS AND STATUS

Introduction

The Dual Cooled Lead Lithium (DCLL) blanket concept, with Pb-Li and He as coolants, is the leading U.S. design for a test blanket module (TBM) for ITER and for a DEMO-type fusion reactor.[1] Reduced activation ferritic-martensitic (RAFM) steels are leading candidates for structural materials, but the rapid dissolution of Fe and Cr in Pb-Li limits their temperature of use to ~475ºC.[2-3]. Structural materials with enhanced creep and compatibility are required for a blanket temperature above 500ºC in a DEMO-type reactor. Nano-structured ferritic (NFA) or ODS FeCr alloys exhibit excellent radiation and creep resistance at high temperature,[4-8] but their compatibility with Pb-Li is similar to RAFM steels. [9-10].

Alumina forming Fe-Cr-Al alloys offer significantly better compatibility with Pb-Li, and only low mass losses were observed for FeCrAl alloys after isothermal compatibility tests at 800ºC.[9] A 550ºC thermal convection loop experiment also showed that only small mass changes were measured for the commercial Fe-21Cr-5Al-3Mo APMT alloy in the hot and cold legs after exposure for 1000h.[11]

ODS FeCrAl alloys are, therefore, very good candidates for structural materials with both creep and compatibility resistance, and new Fe-12Cr-5Al ODS alloys were fabricated at ORNL by ball milling Fe-12Cr-5Al gas atomized powder with Y_2O_3+FeO (125YF alloy) Y_2O_3+ZrO_2 (125YZ), Y_2O_3+HfO_2 (125YH), and Y_2O_3+TiO_2 (125YT) powders. Determination of the mechanical properties and compatibility with Pb-17Li of this first generation of ODS FeCrAl alloys has been completed,[12-16] and the characterization of a second generation of Fe-12Cr-6Al-Zr (Hf) alloys was initiated. For these alloys, Zr and Hf were directly added to the gas atomized powder to improve the homogeneity of the Zr (Hf) dispersion.

Experimental Procedure

Alloy Fabrication

The first generation of ODS FeCrAl alloys were fabricated by ball milling ~1 kg of Fe-12Cr-5Al gas atomized powder with Y_2O_3+FeO (125YF) Y_2O_3+ZrO_2 (alloy 125YZ), Y_2O_3+HfO_2 (alloy 125YH), and Y_2O_3+TiO_2 (alloy 125YT) powders. The powders were degassed for 24 h at 300ºC and extruded at 950ºC

through a rectangular 2x1.5 inch die. Details of the alloy fabrication procedure can be found elsewhere.[12,15]. For the 2nd generation ODS FeCrAl alloys, four different gas atomized powders were purchased from ATI, with targeted compositions of Fe-12Cr-5.5Al-0.55Zr, Fe-12Cr-5.5Al-0.55Zr, Fe-12Cr-5.5Al-0.25Zr-0.13Ti and Fe-10Cr-6Al-0.3Zr. The first two powders were ball milled with Y_2O_3 and extruded the same way as for the 1rst generation ODS FeCrAl alloys (alloy 126ZY and 126ZH). The last two powders as well as a second batch of the Fe-12Cr-5.5Al-0.55Zr powder were also ball milled with Y_2O_3 for 40 h but the resulting powder was extruded at 1000ºC using a smaller extrusion can and a 7/8 inch cylindrical die (alloy 126ZY_10C, 126ZTY and 106ZY_1000ºC). Finally, a second batch of the Fe-10Cr-6Al-0.3Zr powder was extruded at 900ºC using the smaller 7/8 inch die (106ZY_900ºC). The composition of the gas atomized powders and extruded alloys are given in Table 1 and Table 2, respectively.

Table 1. Composition of the gas atomized powders provided by ATI

	Fe	Cr	Al	Zr	C (ppm)	N (ppm)	O (ppm)	S (ppm)	Other
12Cr-5Al / 1rst gen	82.8	12.1	5		31	10	64	<3	
12Cr-5.5Al-0.5Zr	81.8	11.9	5.6	0.54	20	20	140	20	
12Cr-5.5Al-0.25Zr-0.13Ti	81.3	12.2	5.9	0.24	40	20	130	30	0.14Ti
12Cr-5.5Al-1Hf	81.1	12	5.8		130	20	50	40	0.99Hf
10Cr-6Al-0.3Zr	83.1	10	6.4	0.29	40	20	190	30	

Table 2. Composition of some alloys measured by inductively coupled plasma analysis and combustion analysis.

	Alloy	Extrusion Temperature	Die	Fe	Cr	Al	Zr/Hf/Ti	Y	C (ppm)	N (ppm)	O (ppm)	S (ppm)
1rst gen	125YZ	950°C	2"x1.5"	82.8	11.51	4.86	0.3 Zr	0.18	250	161	1920	10
	125YT	950°C	2"x1.5"	82.4	12	4.9	0.2 Ti	0.16	350	140	2220	30
	125YH	950°C	2"x1.5"	82.3	11.68	4.82	0.68 Hf	0.17	220	110	2280	10
2nd gen	126ZY	950°C	2"x1.5"	81.05	11.75	5.69	0.48 Zr	0.2	130	634	1310	30
	126ZH	950°C	2"x1.5"	81.18	11.74	5.75	0.84 Hf	0.2	240	113	1120	40
	126ZY_10C	1000°C	7/8"	81.59	11.54	5.61	0.39 Zr	0.2	150	103	1110	30
	126ZTY	1000°C	7/8"	82.44	11.26	5.4	0.2Zr/0.1ZTi	0.21	330	246	1840	30
	106ZY_10C	1000°C	7/8"	83.41	9.74	6.02	0.28 Zr	0.22	190	580	1290	30
	106ZY_9C	900°C	7/8"	83.34	9.73	6.07	0.28 Zr	0.21	170	919	1300	30

Mechanical testing

Tensile tests were performed using SS-3 type tensile specimens (7.62 mm long and 0.762 mm thick) fabricated with the gage section parallel to the extrusion axis of the ODS FeCrAl alloys. The tensile tests were conducted in an MTS hydraulic frame at temperatures ranging from 25ºC-800ºC and a strain rate of ~$10^{-3}s^{-1}$.

Creep testing was performed using 25 mm long specimens parallel to the extrusion direction and with a 2 x 2 mm gauge section that was 7.6 mm long.

Compatibility tests

Static capsule tests were performed using Mo (inert to Pb-17Li) welded closed inner capsules and type 304 stainless steel (SS) outer capsules to protect the inner capsule from oxidation. The compatibility experiments were conducted at 700ºC for 1000h.[16]

Microstructure characterization

Selected specimens were mounted using conventional metallographic techniques and TEM specimens were prepared using the in situ lift-out method. A Philips model CM200 FEG-TEM/scanning TEM (STEM) with EDS and electron energy loss spectroscopy (EELS) was used for microstructural and compositional analyses. Details of the TEM characterization techniques can be found elsewhere [15-16].

Results

Alloy Characterization

Extensive TEM characterization was conducted on the 125YZ and 125YH alloys, [15] and examples of TEM images and EDS maps are shown in Figure 1 and Figure 2 for alloys 125YZ and 125YH, respectively. The nano precipitates were identified as $Y_3Al_5O_{12}$ (YAG), Al_2O_3 and Zr(C,N) for alloy 125YZ, and $Y_3Al_5O_{12}$(YAG), Al_2O_3, Hf(C,N), $Y_2Hf_2O_7$ and HfO_2 for alloy 125YH. Contrary to what was reported by Yu et al. for an Fe-16Cr-4Al-2W-0.35Y_2O_3 + Zr alloy, no $Y_2Zr_2O_7$ precipitates were observed in alloy 125YZ.[17] The presence of HfO_2 particles in alloy 125YH indicates that the 40h ball-milling step was likely insufficient to break down completely the HfO_2 powder and drive Hf into solution in the Fe matrix.

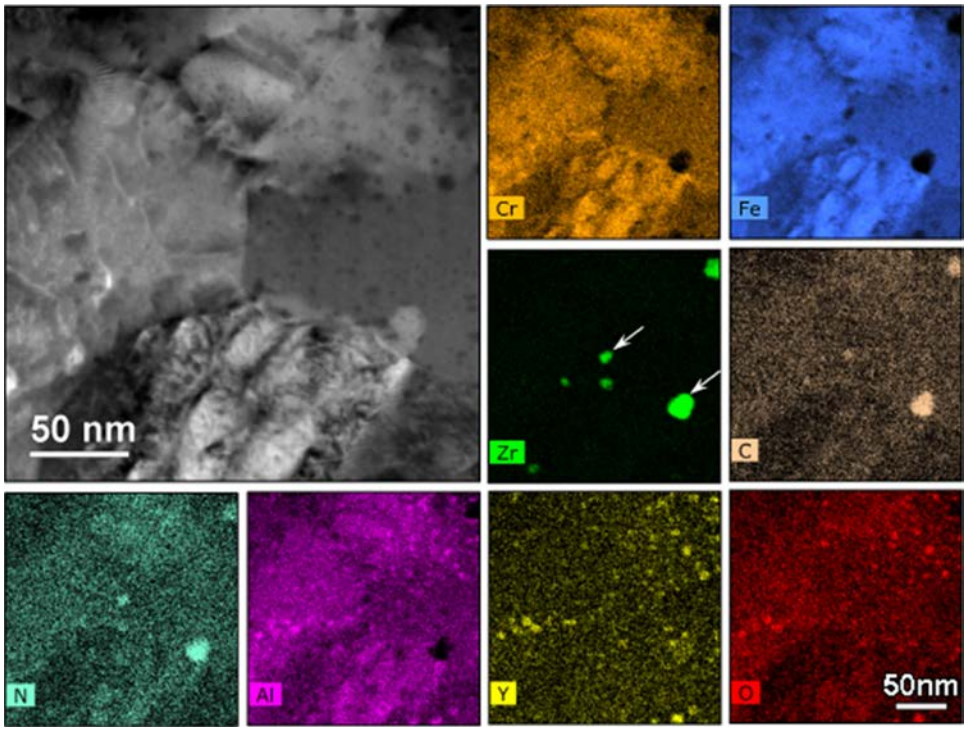

Figure 1. HAADF-STEM image of the 125YZ as-extruded (lift-out FIB) alloy with corresponding EDS elemental maps of Cr, Fe, Zr, C, N, Al, Y, and O.[15]

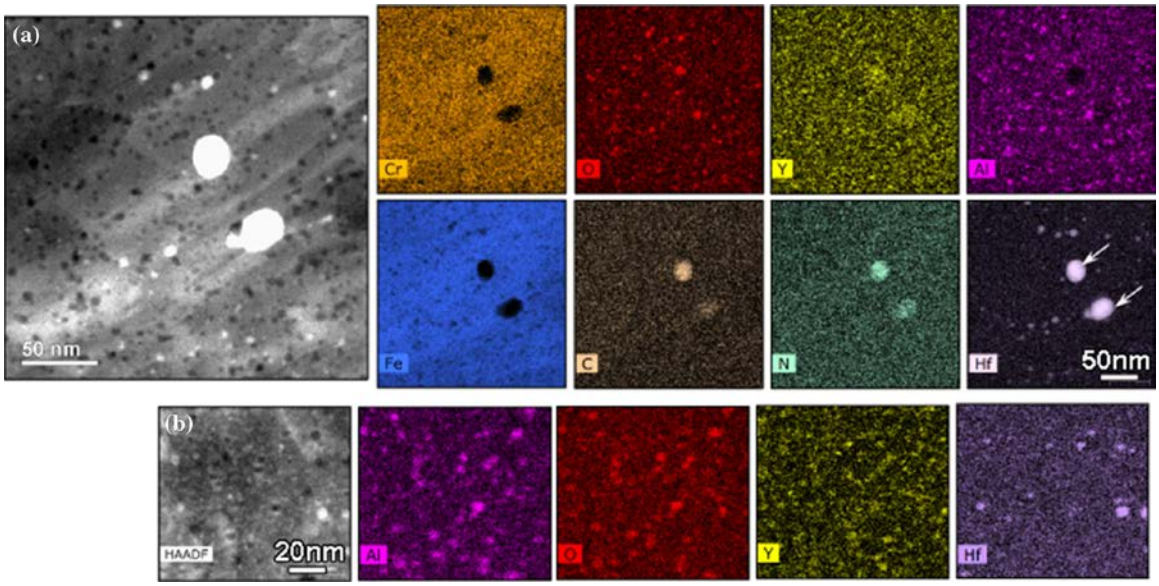

Figure 2. a) HAADF-STEM image of the as-extruded 125YH (lift-out FIB) alloy, b) corresponding EDS elemental maps of Al, O, Y, Cr, Hf, C, N, and Fe.[15]

Creep testing

Creep tests were conducted at 800ºC with an applied stress of 100 MPa, and the results for alloy 125YZ, 125YH and 125YT are summarized in Figure 3a. Alloy 125YZ performed significantly better than alloy 125YH and 12YZ, and the two 125YZ tests were interrupted before failure after respectively 1000 h and 6000 h of exposure for microstructure characterization. The microstructure of alloy 125YH was less homogeneous than the microstructure of alloy 125YZ, which might explain the early creep failures for alloy 125YH, and alloy 125YT seems less stable at temperature above 800ºC than alloy 125YZ.

Figure 3b compares the creep properties of alloy 125YZ at 700 and 800ºC with the creep properties of the former commercial alloy PM2000 and new ODS FeCrAl alloys developed in Japan.[18,19] At 800ºC, the 6000 h lifetime corresponds for alloy PM2000 to a stress of ~45 MPa, whereas alloy 125YZ did not fail after 6000 h with an applied stress of 100 MPa. At 700ºC, the best performing Japanese alloy, Fe-16Cr-4Al-2W-Y+Zr, was expected to reach the ~10,000 h lifetime limit for an applied stress of 120 MPa. The 125YZ alloy exhibited a significantly better creep behavior with one test at 700ºC, 140 MPa still running after ~16,000 h.

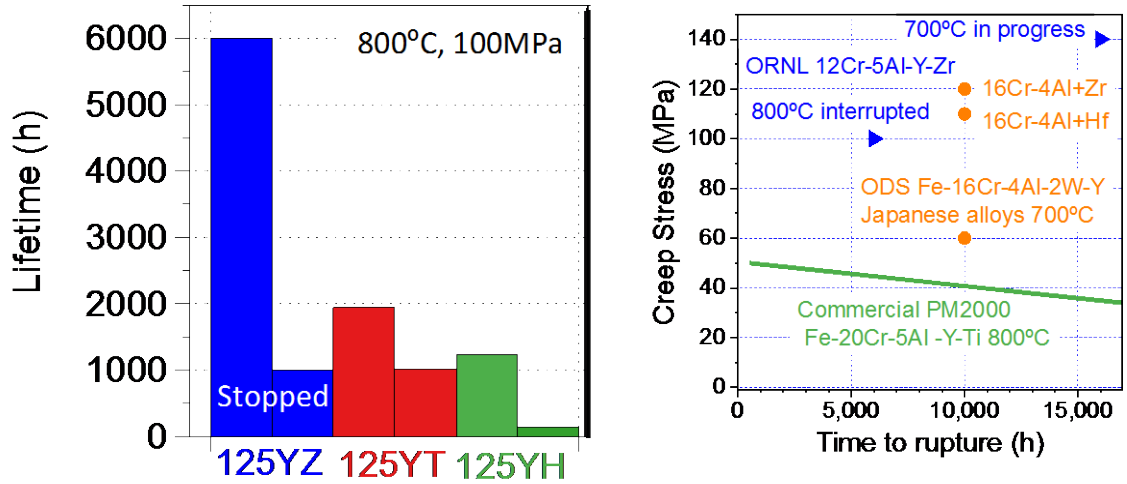

Figure 3. a) Creep lifetime at 800ºC, 100 MPa for the 125YZ, 125YT and 125YH alloys, b) comparison of the creep performance at 700-800ºC between alloy 125YZ and other ODS FeCrAl alloys.

Compatibility tests

The 125YZ, 125YH and 125YT alloys were exposed to Pb-17Li in static capsule experiments for 1000 h, and the detailed analysis of these results can be found elsewhere. [16] Very limited mass changes were measured for all the alloys with the formation after 1000 h of a thin $LiAlO_2$ layer ~0.5 to 1.5 um thick.

Example of the oxide scale TEM characterization is shown in Figure 4 for alloy 125YZ. Void formation was observed both in the scale and at the alloy/scale interface, and Zr-rich precipitates were identified in the scale. Similarly, voids formation, and Hf and Ti-rich precipitates were observed in the scale formed on alloy 125YH and 125YT, respectively.

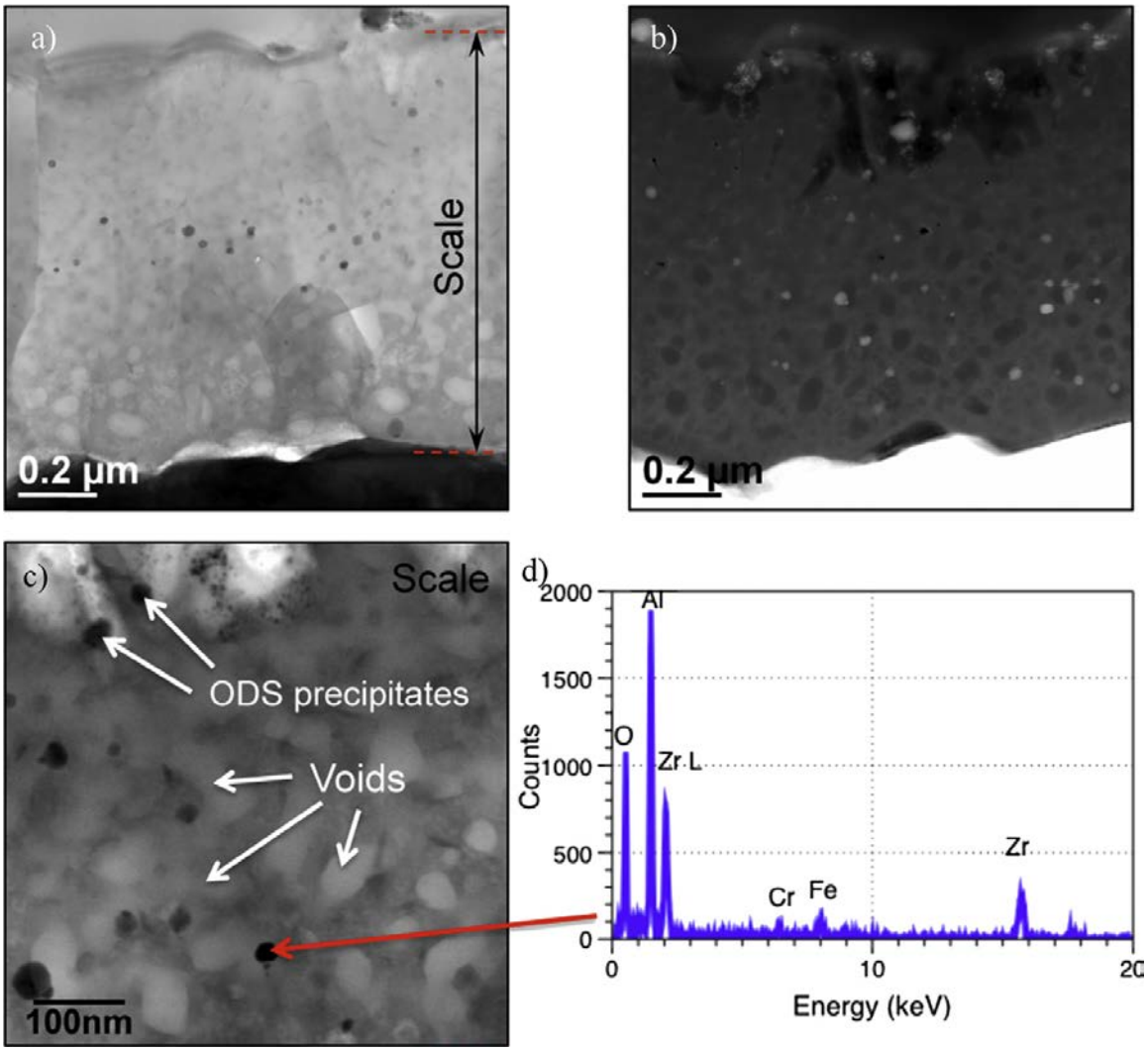

Figure 4. Cross-section of the oxide formed on 125YZ after exposure for 1000h at 700ºC in static Pb-17Li, (a,c) BF-STEM images, b) HAADF-STEM images, d) EDS point spectrum generated from a particle within the oxide.

2nd generation ODS alloys

The second generation of ODS FeCrAl alloys was fabricated by ball-milling FeCrAl+ Zr, Hf or Zr+Ti powders with Y_2O_3 powder (Tables 1 and 2). The goal was to improve the alloy homogeneity and the control of the O content in the alloy. The tensile properties of the 2nd gen 126YZ and 126YH alloys are compared in Figure 5 with the properties of the 1st generation 125YZ and 125YH alloys. At temperature below 400ºC, alloy 126YZ exhibited very high strength but no ductility, whereas the tensile strength of alloy 126HY was lower than the strength of the 1st gen ODS FeCrAl alloys.

The tensile properties of several 2nd generation alloys extruded through a smaller 7/8 inch die are again compared with the tensile properties of alloy 125YZ in Figure 6. All the 2nd generation alloys extruded at 1000ºC exhibited very similar tensile behavior, with lower tensile strength but higher ductility than alloy 125YZ. These results indicate that small variations in Zr concentration or addition of both Zr and Ti have limited effects on the alloy tensile properties. On the contrary, extrusion conditions seem to have a

tremendous impact on the alloy tensile properties, with the 126ZY and 126ZY_10C alloys, fabricated with the same ball milled powder, showing very different tensile behaviors.

Finally, it is worth noting that the 106ZY_9C alloy extruded at 900ºC showed similar strength but better ductility than the 1st generation 125YZ alloy. This alloy is a promising candidate for structural applications in fusion reactors and specimens have been machined to initiate creep testing at 700 and 800ºC. Optimization of the processing conditions to enhance the creep performance and fabricability of ODS FeCrAlYZr alloys is ongoing.

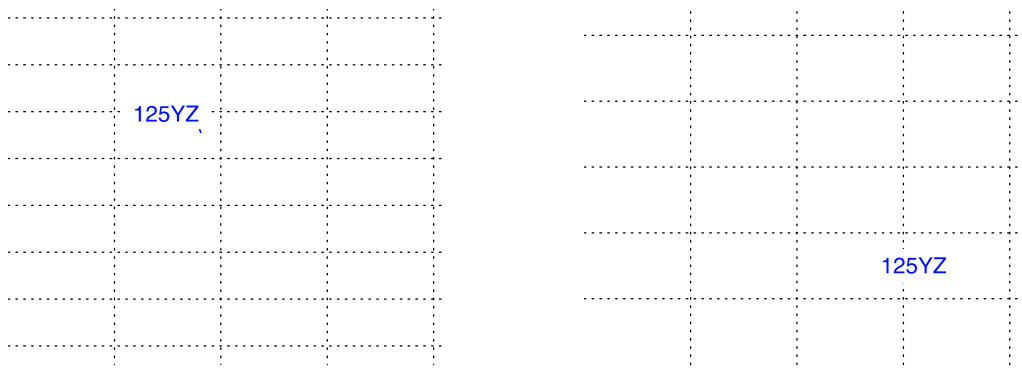

Figure 5. Tensile properties of the 1st generation 125YZ and 125YH alloys, and 2nd generation 126YZ and 126HY ODS alloys, a) Ultimate tensile strength, b) Plastic deformation.

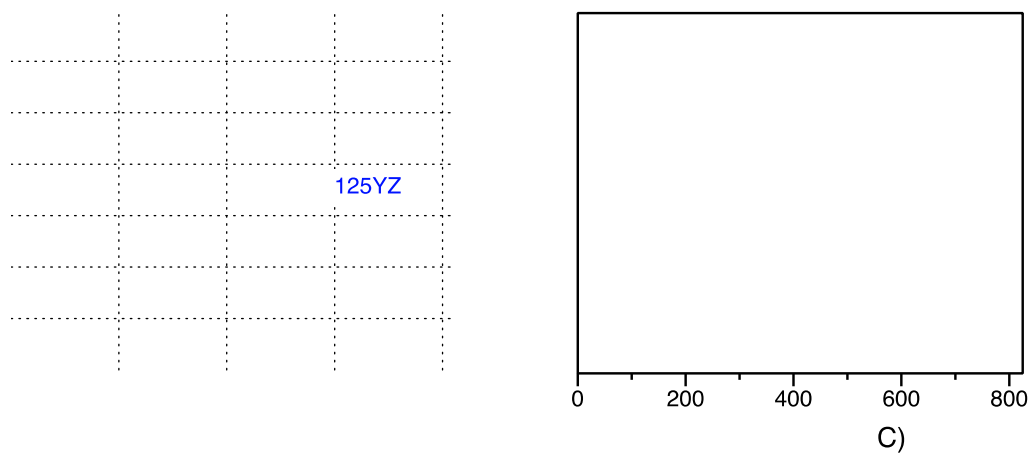

Figure 6. Tensile properties of the 1st generation 125YZ alloy, and several 2nd generation ODS FeCrAl alloys extruded at 1000ºC or 900ºC, a) Ultimate tensile strength, b) Plastic deformation.

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