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Development of oxide dispersion strengthened ferritic steels for fusion

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Abstract

An oxide dispersion strengthened (ODS) ferritic steel with high temperature strength has been developed in line with low activation criteria for application in fusion power systems. The composition Fe–13.5Cr–2W–0.5Ti–0.25Y_2O_3 was chosen to provide a minimum chromium content to insure fully delta-ferrite stability. High temperature strength has been demonstrated by measuring creep response of the ODS alloy in uniaxial tension at 650°C and 900°C in an inert atmosphere chamber. Results of tests at 900°C demonstrate that this alloy has creep properties similar to other alloys of similar design and can be considered for use in high temperature fusion power system designs. The alloy selection process, materials production, microstructural evaluation and creep testing are described.

1. Introduction

The oxide dispersion strengthened (ODS) ferritic steel called MA 957 [1], produced by mechanical alloying, has received international consideration for fuel cladding applications in liquid metal fast breeder reactors [2]. The alloy shows excellent long term microstructural stability in irradiation environments [3] and is expected to retain superb high temperature strength. This MA 957 alloy has the composition Fe–14Cr–1Ti–0.25Mo–0.25Y_2O_3. The microstructure consists of a metal matrix with uniformly distributed Y_2O_3 dispersoids on the order of 5 nm in diameter. It also contains a highly elongated subgrain structure which is introduced by thermo-mechanical processing. Based on the performance demonstrated to date, this technology should be considered for first wall applications of a fusion reactor. Therefore, an effort has been initiated [4–7] to consider the use of mechanically alloyed ODS alloys for fusion by altering the alloy composition to be in line with low activation criteria, and determining the creep response of the ODS alloy.

2. Experimental procedure

The starting powder compositions and the maximum mesh sizes are shown in Table 1. A Spex 8000 shaker mill was used for the mechanical alloying. The alloy was optimized by studying the phase transformations of the powders which are mechanically alloyed and then annealed at 1000°C for 1 h, using a Perkin-Elmer DTA 7 differential thermal analyzer (DTA). The optimization of the composition was achieved by studying seven ODS steels, Fe–(5, 9, 10, 11, 12, 13, 13.5) Cr–2W–0.5Ti–0.25Y_2O_3, produced by mechanical alloying. The milled powders were also characterized in a Siemens D 5000 X-ray powder diffractometer to understand the structural evolution during milling. Low carbon steel tubing with 1 in. ID × 0.14 in. wall thickness × 13 in. overall length was used for HIP canning. The cans were pumped down to 20 mtorr and then baked at 400°C for 30 h and heat crimped before HIPPing. The hot isostatic pressing was then carried out at 950°C and 210 MPa by IMT, Andover, MA. The outside diameter of the HIPPed billets (including the can) was 7/8 in. After soaking at 950°C for 1 h following HIPPing the billets were hot swaged in a hot swaging unit. The outer diameter of the hot swaged materials was 1/2 in. The cans were opened after

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Table 1
Powder specifications

Powders	Size (mesh)	Composition (wt%)
Fe	−325	99.4
Fe–Cr master	−200	Fe–73.53 Cr
Fe–Ti master	−100	Fe–40.53 Ti
W	−200	99.9
Y_2O_3	−325	99.9

hot swaging. The inner diameter of the ODS material was found to be 5/16 in. Hardness measurements of the ODS compacts were done in a Tukon hardness tester. Thin foils were made from the compact and characterized in JEOL 1010 and 2010 F transmission electron microscopes (TEM). The chemical assay of the milled powders was done by Crucible Research, Pittsburgh, PA. A SATEC creep frame, with 20:1 lever arm, fitted with an inert atmosphere Al_2O_3 retort, was used for creep testing. Strain was measured using an extensometer attached to a Linear Variable Differential Transformer (LVDT) located away from the high temperature zone.

Specimens were made from two batches of rod material. Specimens were 1.50 in. long, with a gauge section 1.020 in. long and 0.170 in. in diameter, with grip sections 0.250 in. in diameter. Specimen temperature was continuously monitored during heat up and throughout the test with two type-K thermocouples, one attached at the top and the other at the bottom of the specimen gauge length. The temperature gradient between the top and bottom of the specimen was minimized during the test, and did not exceed 2°C. The average specimen temperature was maintained to within ±20°C of the desired test temperature for the duration of the test. Specimens were heated to the desired test temperature for the duration of the test. Specimens were heated to the desired test temperature in 2–4 h and then were held for about 1 h at the test temperature to equilibrate and stabilize the specimen temperature prior to loading.

3. Results and discussion

3.1. Alloy optimization

The X-ray diffraction patterns of all seven alloys indicated the presence of only the α-Fe solid solution after 10 h of milling. Fig. 1 shows the X-ray diffraction patterns of the Fe–13.5Cr–2W–0.5Ti–0.25Y_2O_3 (ODS 13.5 Cr) powder as a function of milling time. All seven ODS steel powders produced by mechanical alloying were annealed at 1000°C for 1 h prior to the phase transformation studies in a DTA. The DTA plots of the ODS steel powders for 5, 13 and 13.5 Cr milled for 10 h (Fig. 2) indicates no austenite transformation only in the ODS 13.5 Cr steel. The interstitial impurity content (wt%) of the ODS 13.5 Cr steel powder milled for 10 h was: carbon – 0.055; oxygen – 0.855; and nitrogen – 0.284.

The variations in composition studied covered 5–13.5 Cr. It can be noted that in the pure Fe–Cr system the austenite loop ends at a composition of 12.7 Cr. The presence of 2% W and 0.5% Ti in the α-Fe solid solution should further suppress the austenite loop to about 10.75 Cr (calculated from chromium equivalent formula [8]) and hence the transformation to austenite should not be observed in the alloys with Cr > 10.75%. However, the austenite formation was observed by DTA in

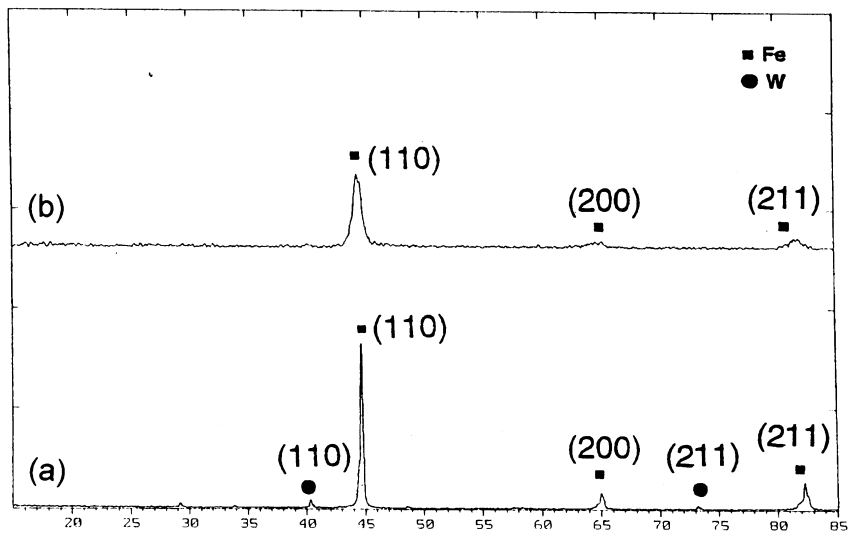

Fig. 1. X-ray diffraction patterns of the ODS–13.5Cr powder as a function of milling time.

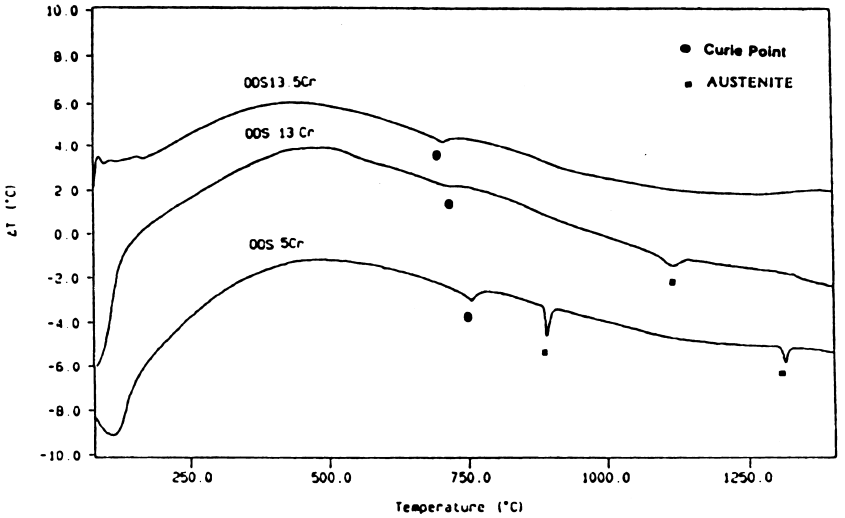

Fig. 2. DTA plots of the ODS–5, 13 and 13.5 Cr powders milled for 10 h showing no austenite transformation only in ODS–13.5Cr.

the ODS (11, 12, 13) Cr alloys. The presence of nitrogen and carbon which act as austenite stabilizers are probably responsible for the austenite transformations in ODS (11, 12, and 13) Cr alloys.

3.2. Characterization

Metallographic sections of the as-swaged material in both the transverse and longitudinal directions were prepared and the results are shown in Fig. 3. The micrograph reveals the elongated grain structure in the longitudinal direction. No porosity was observed in the material. A calculation of the bulk density from the machined dimensions showed the material to be 100% dense. The hardness of the as-swaged material was measured to be 65 R_c. The TEM micrograph in Fig. 4 shows the elongated grain structure and also the presence of very fine dispersoids of sizes less than 10 nm in the ferrite matrix. The as-swaged material was annealed at 800°C, 900°C, 1000°C, 1100°C and 1200°C for 1 h in order to examine the change in the hardness with annealing temperature.

A plot of hardness versus annealing temperature in Fig. 5 showed a slight decrease in the hardness value with an increase in the annealing temperature. The plot in Fig. 5 clearly indicates that the hardness remains very high even following a 1200°C anneal.

The as-swaged material shows an elongated microstructure both in optical and transmission electron microscopy typical of hot worked ferritic ODS alloys. Analytical microscopy also indicated high homogeneity in the material. The hardness of the swaged material was very high due to the high dislocation density in the material. High hardness value was retained after 1 h exposure of the as-swaged material at 1200°C indicating a high thermal stability of the microstructure. Therefore,

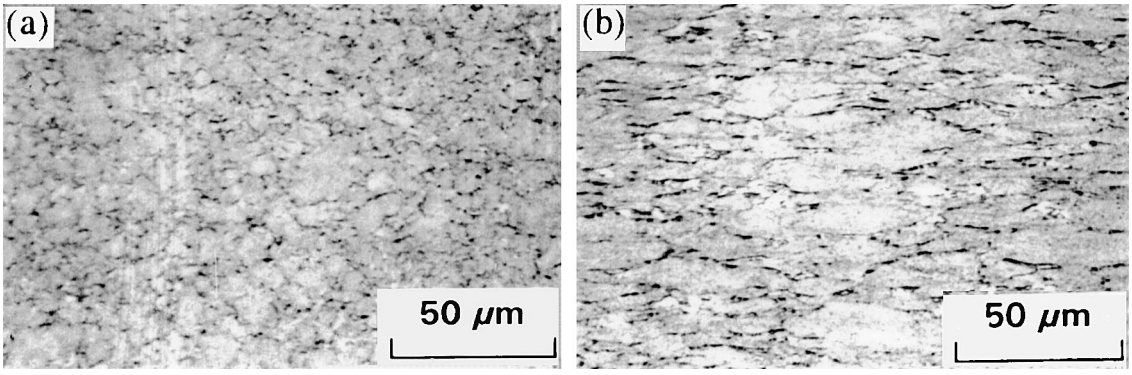

Fig. 3. Polished and etched (Vilella's reagent) sections of as-swaged ODS–13.5 Cr material are shown in (a) transverse and (b) longitudinal directions.

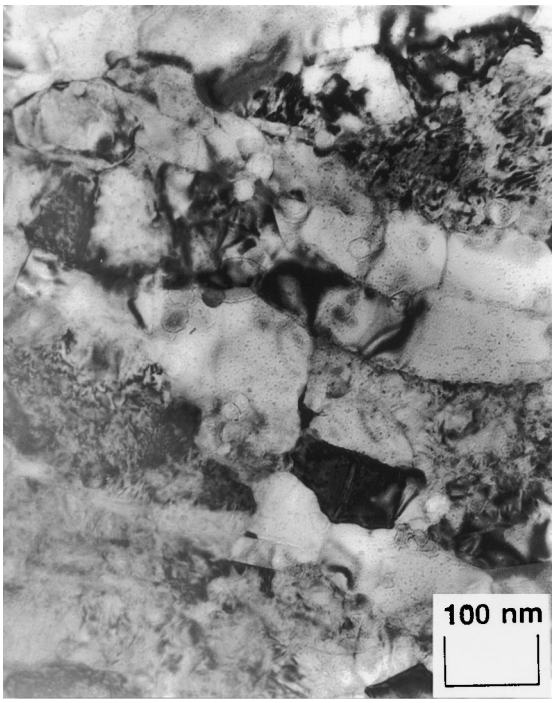

Fig. 4. Elongated grains in the longitudinal direction of the ODS–13.5Cr as-swaged material.

excellent high temperature creep response can be anticipated.

3.3. Creep test results

Specimens were tested in argon at 650° and 900°C at stresses ranging from 90 to 350 MPa with specimens being tested to failure in all cases. Table 2 provides test parameters, elongation and rupture time for each specimen. Figs. 6 and 7 show creep plots of strain versus time for two test specimens from sample #1. About 36% strain was observed during creep test no. 1 which failed after 520 h (Fig. 6). Creep test no. 2 which lasted 22 h (Fig. 7), however, showed 25% strain before failure. Figs. 8 and 9 show the plots of creep strain versus time for two test specimens from sample #2. Creep test no. 3 which lasted for about 2 h (Fig. 8) showed only 10% elongation. Creep test no. 4 which lasted for about 14 h showed only 6% elongation before failure.

3.4. Fractography of creep specimens after failure

Scanning electron microscopy (SEM) examination demonstrated necking during creep rupture testing of sample #1/test #1 at 900°C, 90 MPa. The necking clearly indicated good ductility for sample #1. However, SEM examination of sample #2/test #1 showed no

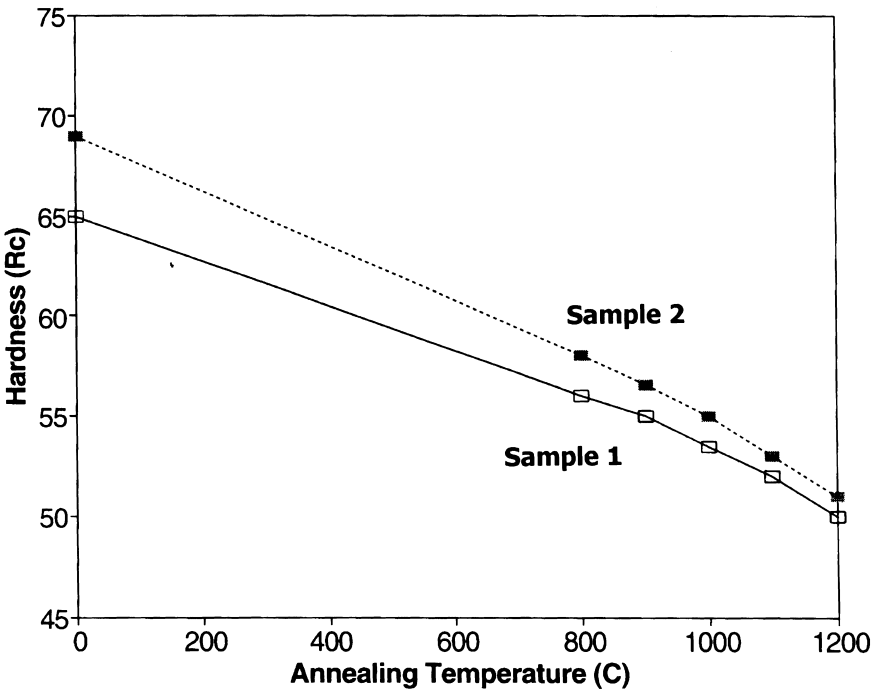

Fig. 5. A plot of hardness versus annealing temperature (1 h anneals) indicating a slight decrease in the hardness with temperature.

Table 2
Creep test parameters and test results

No.	Test	Stress (MPa)	Temp. (°C)	Elong. (%)	Rupture time (h)
1	1	90	900	36	520
	2	150	900	25	22
2	3	150	900	10	2
	4	350	650	6	14

necking during creep rupture testing at 900°C, 150 MPa, which confirmed the reduced ductility for sample #2.

3.5. Comparison of creep response

The creep test performed on sample #1 at 900°C at 90 MPa indicates a 520 h rupture life with an elongation of 36% before failure (Fig. 6). Therefore, good strength and ductility can be obtained in this class of alloys at high temperature. Comparison of the creep rupture results between sample #1 and sample #2 indicates that the creep ductility in sample #2 is similar to that observed in sample #1. Therefore good reproducibility exists between different batches of powder [9].

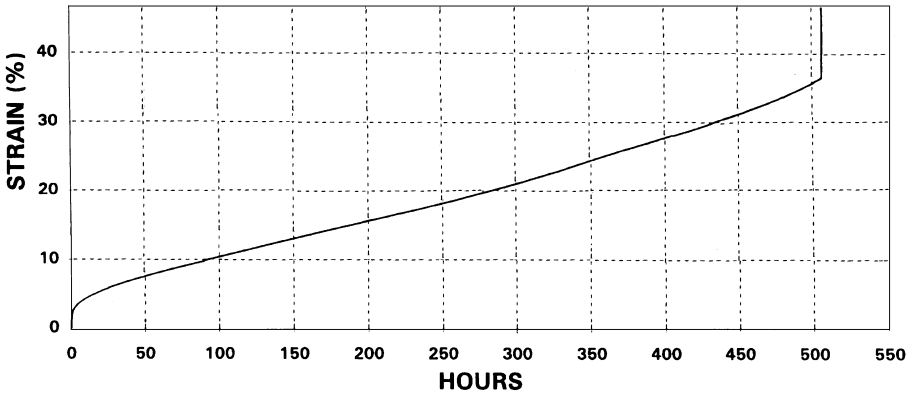

Fig. 6. Creep strain versus time in Sample #1 at 900°C and 90 MPa.

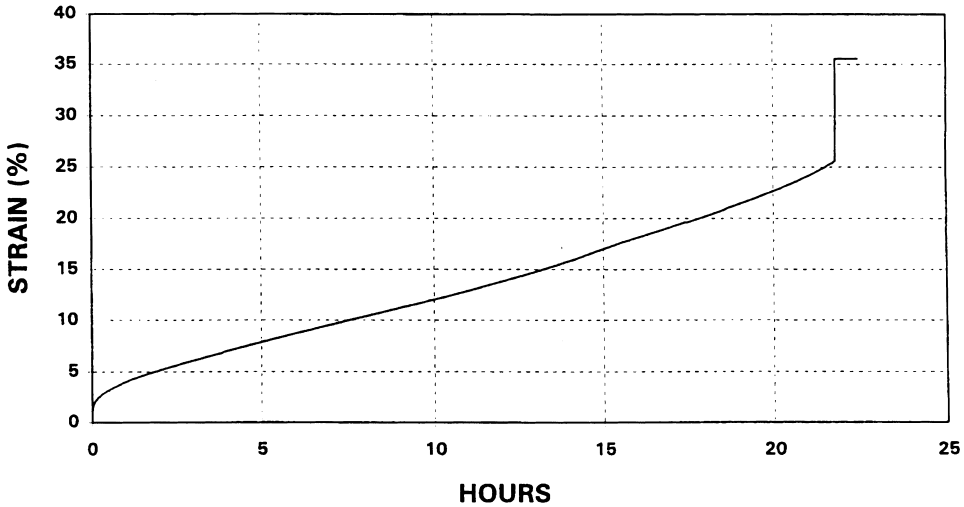

Fig. 7. Creep strain versus time in Sample #1 at 900°C and 150 MPa.

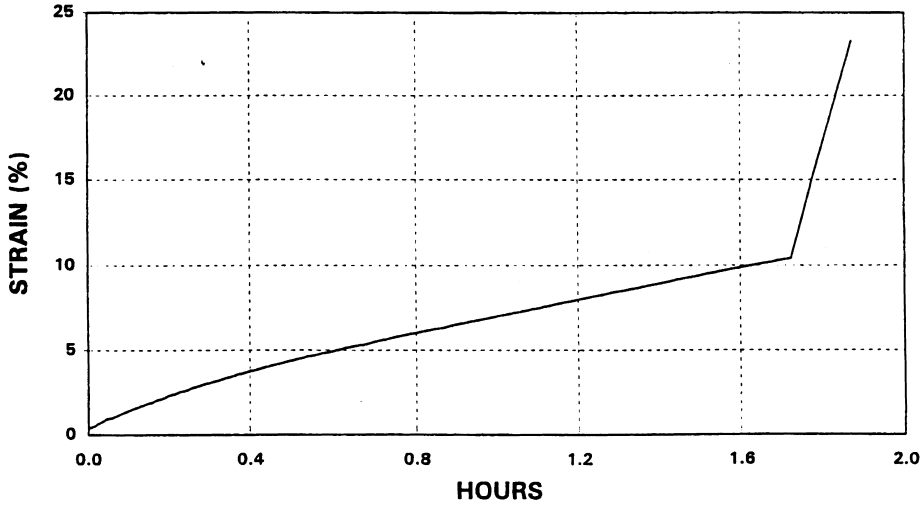

Fig. 8. Creep strain versus time in Sample #2 at 900°C and 150 MPa.

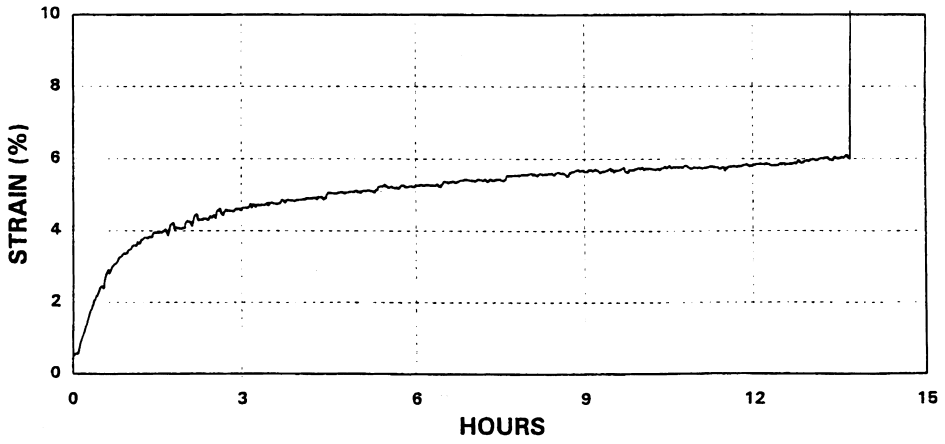

Fig. 9. Creep strain versus time in Sample #2 at 650°C and 350 MPa.

The merit of the new low activation ODS alloy can be demonstrated using a Larson–Miller plot. Fig. 10, a Larson–Miller plot of stress rupture data plotting rupture stress as a function of temperature and time to rupture shows a summary of creep rupture values of the present ODS alloy (sample #1 and sample #2), MA 957 [1], MA 956 [10], two similar ODS steels developed in Japan by Asabe [11] and Ukai [12], a Belgian ODS steel [13], and HT-9 martensitic steel [14]. From this it can be seen that despite limited data, the new low activation alloy compares favorably with similar ODS ferritic alloys MA956 and MA957, which are far superior to martensitic steels but somewhat below that from Asabe and Ukai. The very fine and uniform dispersion is mainly responsible for the superior creep resistance of the present ODS alloy.

4. Conclusions

A low-activation grade ODS ferritic steel has been optimized (Fe–13.5Cr–2W–0.5Ti–0.25Y_2O_3) and successfully manufactured by mechanical alloying. High material homogeneity was observed by electron microscopic study. Hardness remains very high even following a 1200°C annealing treatment indicating a highly stable microstructure. The optimized low activation grade ODS ferritic steel has been tested in uniaxial creep in

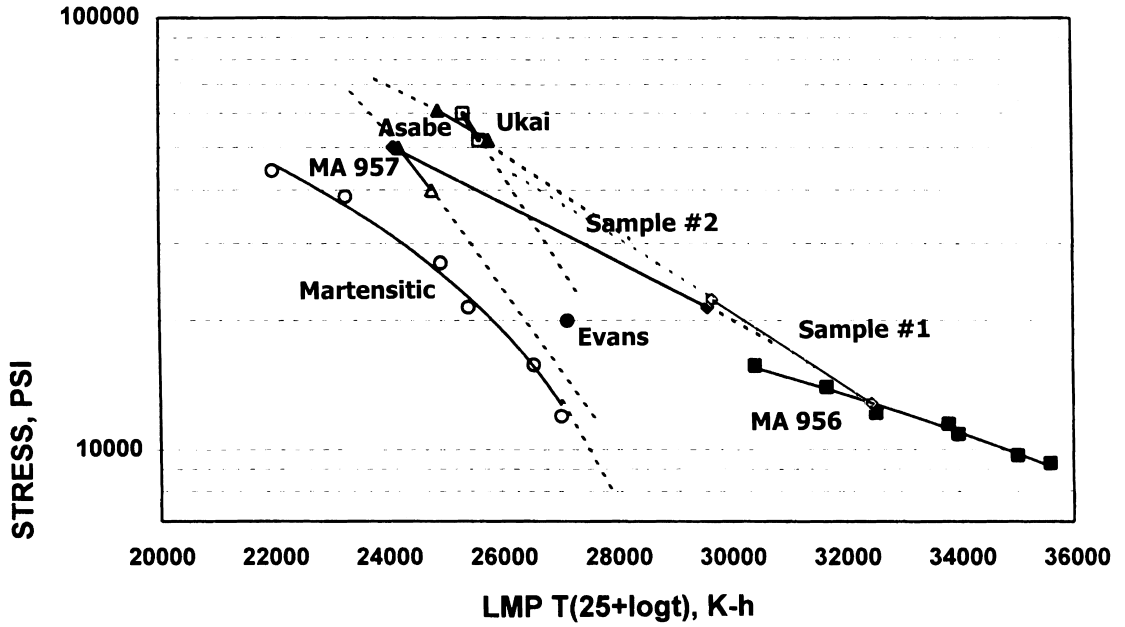

Fig. 10. Larson–Miller plot comparing the creep strength of the new ODS ferritic steel with those of similar steels.

order to provide comparison with similar composition. Results of tests at 900°C demonstrate that this alloy has creep properties similar to other alloys of similar design and can be considered for use in high temperature fusion power system designs.

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