ELSEVIER

Journal of Nuclear Materials 283–287 (2000) 697–701

journal of nuclear materials

www.elsevier.nl/locate/jnucmat

A potential new ferritic/martensitic steel for fusion applications

R.L. Klueh [a,*,1], N. Hashimoto [a], R.F. Buck [b], M.A. Sokolov [a]

[a] *Oak Ridge National Laboratory, Metals and Ceramics Division, P.O. Box 2008, MS 6376, Oak Ridge, TN 37831-6376, USA*

[b] *Advanced Steel Technology, 6402 Ten Point Circle, Trafford, PA 15085, USA*

Abstract

The A-21 steel is an Fe–Cr–Co–Ni–Mo–Ti–C steel that is strengthened by a fine distribution of titanium carbide (TiC) precipitates formed by thermomechanical treatment. Transmission electron microscopy of the A-21 reveals a high number density of small TiC particles uniformly distributed in the matrix. Below ≈ 600°C, the strength of A-21 is less than the average value for conventional Cr–Mo or reduced-activation ferritic/martensitic steels. However, the strength is greater above 600°C. The Charpy impact properties of A-21 are comparable to those of the conventional and reduced-activation steels. Due to the fine TiC particles in the matrix, the creep-rupture properties of A-21 are superior to those of conventional Cr–Mo or reduced-activation Cr–W steels. Although the composition of the A-21 is not applicable for fusion because of the cobalt, the innovative production process may offer a route to an improved steel for fusion. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

High-chromium ferritic/martensitic steels, such as the conventional Cr–Mo steels (modified 9Cr–1Mo and Sandvik HT9) or the reduced-activation Cr–W steels (F82H, ORNL 9Cr–2WVTa, and JLF-1), being considered for a fusion reactor first wall and blanket structure would limit the upper operating temperature to 550–600°C. One way suggested to increase this limit to 650°C or higher and still maintain the advantages inherent in ferritic/martensitic steels (e.g. high thermal conductivity and low swelling) is to use oxide dispersion-strengthened (ODS) steels. Elevated temperature strength of these steels is obtained through microstructures that contain a high density of small Y_2O_3 or TiO_2 particles dispersed in a ferrite matrix.

Unfortunately, production of ODS steels involves complicated and expensive powder metallurgy and mechanical alloying procedures that usually involve extrusion. The directionality deriving from these processing procedures generally results in anisotropic mechanical properties.

Obviously, a ferritic or martensitic steel that could be used at 650°C and above that could be formed by more conventional steel processing techniques would result in a cheaper product than ODS steels produced by powder metallurgy/mechanical alloying procedures. Furthermore, with such a processing technique, it should be easier to produce a non-directional microstructure, which has been a problem for the ODS steels. Such an experimental steel, called A-21, has been developed [1]. Although A-21 might not be directly applicable for fusion, if the properties are satisfactory, the production technique used for A-21 may be applicable to produce an acceptable steel for fusion.

2. Experimental procedure

The A-21 steel is an Fe–9.5Cr–3Co–1Ni–0.6Mo–0.3Ti–0.07C steel (all compositions are in wt%) [1]. The 181-kg heat of steel used for this study was produced as 17.5-mm thick plate that was austenitized at >1100°C to dissolve the carbides. Austenitization was followed by cooling to an intermediate temperature (700–1000°C), where the steel was hot worked in the austenitic condi-

---

[*] Corresponding author. Tel.: +1-865 574 5111;fax: +1-865 574 0641.

E-mail address: ku2@ornl.gov (R.L. Klueh).

[1] Research sponsored by the Office of Fusion Energy Sciences, US Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

0022-3115/00/$ - see front matter © 2000 Elsevier Science B.V. All rights reserved.
PII: S 0 0 2 2 - 3 1 1 5 (0 0) 0 0 1 1 3 - 6

tion. After hot-working, the steel was cooled to ambient temperature to transform the matrix to martensite. Finally, the steel was tempered in the range 650–750°C for 1 h. Miniature tensile and Charpy specimens were machined from the tempered steel plate.

Tensile specimens 44.5-mm long with a reduced gage section of $20.3 \times 1.52 \times 0.76$ mm^3 were machined from the tempered plate with gage lengths parallel to the rolling direction. Tests were at room temperature to 700°C in vacuum at a nominal strain rate of $\approx 4 \times 10^{-4}$ s^{-1}. One-third-size Charpy specimens $3.3 \times 3.3 \times 25.4$ mm^3 with a 0.51-mm-deep 30° V-notch and a 0.05–0.08-mm-root radius were machined from the plate along the rolling direction with the notch transverse to the rolling direction (LT orientation). Charpy tests were carried out in a pendulum-type impact machine specially modified to accommodate subsize specimens [2]. Details of the test procedure have been published [3–5].

Properties of A-21 were compared with properties previously determined on modified 9Cr–1Mo (9Cr–1MoVNb) steel, a Fe–9Cr–1Mo–0.2V–0.07Nb–0.03N–0.1C steel [6], Sandvik HT9 (12Cr–1MoVW), a Fe–12Cr–1Mo–0.5W–0.5Ni–0.25V–0.2C steel [6], ORNL 9Cr–2WVTa, a Fe–9Cr–2W–0.25V–0.07Ta steel [7], and F82H, a Fe–7.5Cr–2W–0.2V–0.02W steel [7]. These steels, were tested in their standard normalized-and-tempered conditions [6,7].

The A-21 steel was examined by optical and transmission electron microscopy (TEM). Standard 3-mm diameter TEM disks were machined from the center of the 17.5-mm plate. Disks were thinned using an automatic tenupole electropolishing unit and were examined using a JEM-2000FX (LaB_6) microscope.

3. Results

3.1. Microstructure

Optical microscopy indicated that the steel had a 100% tempered martensite structure with a prior austenite grain size of 5–15 μm. TEM revealed a subgrain structure typical of a tempered martensite structure. The subgrains contained a high number density of precipitates (Figs. 1(a) and (b)) uniformly distributed with no indication of denuded zones near lath or prior austenite grain boundaries. Although some precipitates formed on boundaries, the number density and size of precipitates on the boundaries were not substantially different from those in the matrix. Essentially all the precipitates in the matrix formed on dislocation lines (Figs. 1(c) and (d)).

Diffraction measurements and Moiré fringe measurements indicated the precipitates were titanium carbide (TiC). No other precipitates were observed. There were no indications of strain fields around the precipitates. The TiC particle size varied from about 5 to 20 nm, with the average size of about 9.3 nm. The total number density was estimated to be 4.7×10^{21} m^{-3}.

3.2. Tensile properties

Fig. 2 shows the yield stress of A-21 compared with average properties of modified 9Cr–1Mo steel [8]. The 9Cr–1MoVNb steel is stronger than A-21 at the lowest test temperatures, but above ≈ 600°C, the A-21 becomes stronger than the 9Cr–1MoVNb steel. Similar results were observed for the ultimate tensile strength. The ductilities are also similar, with the values for the A-21 being somewhat higher than for the average 9Cr–1MoVNb steel [8]. Reduced-activation steels (e.g., ORNL 9Cr–2WVTa, F82H, JLF-1) have tensile properties similar to modified 9Cr–1Mo, so a similar comparison applies for these steels.

3.3. Charpy impact properties

Charpy impact properties for A-21 steel are given in Table 1 along with those of modified 9Cr–1Mo [6], Sandvik HT9 [6], ORNL 9Cr–2WVTa [7], and F82H [7]. The ductile–brittle transition temperature (DBTT) for A-21 at half the upper-shelf temperature was similar to that of the modified 9Cr–1Mo steel and the Sandvik HT9 and somewhat less than for the 9Cr–2WVTa and F82H, while the upper-shelf energy for the A-21 is higher than that of the other steels.

4. Discussion

Since A-21 steel contains 3% cobalt, it would not be an acceptable structural material for a neutron environment, such as a fusion power plant, because transmutation of cobalt would produce a highly radioactive structure. The objective of these studies was to determine if the A-21 steel possessed the properties required for higher operating temperatures of a fusion plant than are possible with conventional Cr–Mo steels or reduced-activation Cr–W steels. If higher operating temperatures are possible for A-21, then it would appear reasonable to seek the development of an acceptable composition with the process used to produce A-21.

At present, the best candidate ferritic/martensitic steels available to raise the operating temperature over that possible with the conventional and reduced-activation steels appear to be the ODS steels. Although ODS steels have been produced for some time, they still have problems with anisotropic properties that originate from the production techniques. Mechanical alloying and powder metallurgy production techniques used for ODS steels are expensive, and given the more conventional

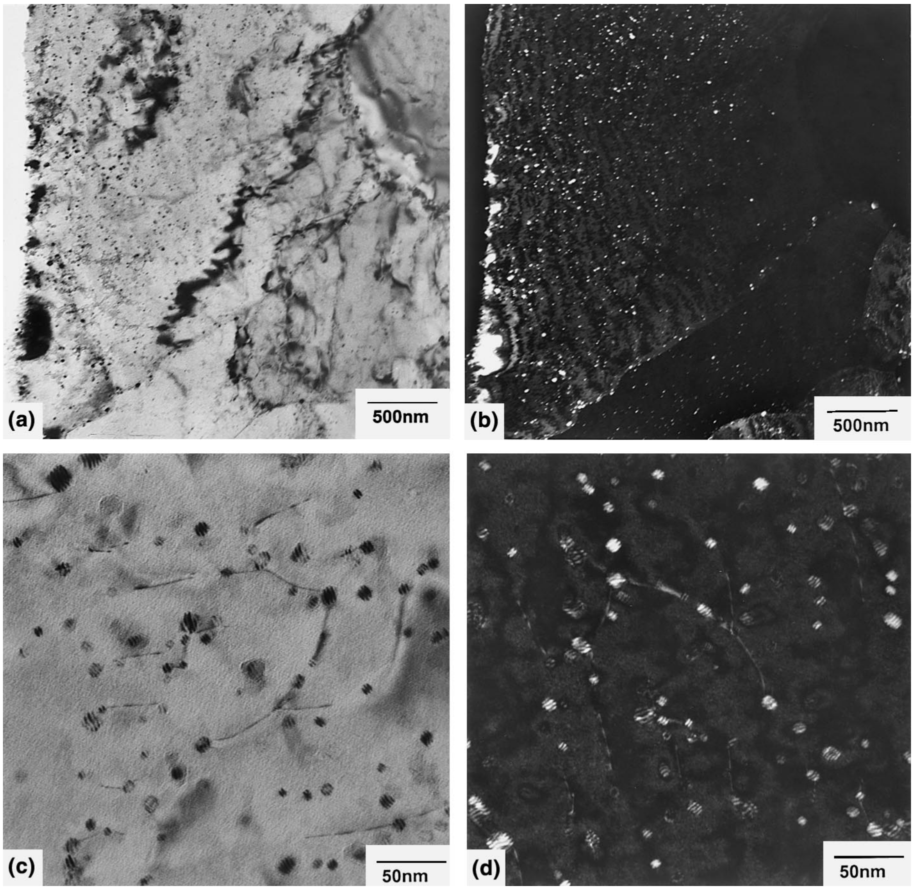

Fig. 1. Transmission electron micrograph showing high number density of TiC precipitates in matrix in: (a) bright field and (b) dark field, and showing precipitates on dislocations in (a) bright field and (b) dark field.

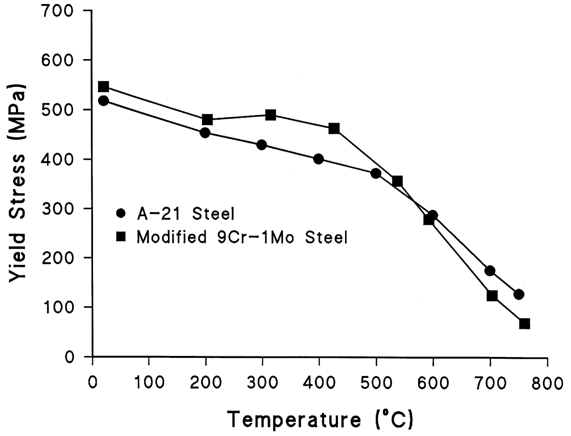

Fig. 2. Tensile properties of A-21 steel compared to modified 9Cr–1Mo steel.

processing used for the A-21 steel, an A-21-type steel would have significant advantages for fusion applications, as well as many other applications.

The mechanical properties of A-21 steel appear appropriate for fusion applications. Irradiation embrittlement, which causes reduced toughness and is observed as an increase in DBTT and a decrease in USE in a Charpy impact test, causes the most concern for ferritic/martensitic steels. Generally, steels with a low DBTT before irradiation have a low value after irradiation. The modified 9Cr–1Mo steel, which has a DBTT similar to A-21 (Table 1), showed a relatively small increase (45°C) in DBTT when irradiated in the Fast Flux Test Facility at 365°C [6]. The A-21 had unirradiated properties similar to modified 9Cr–1Mo. Since the shift in DBTT is caused by irradiation hardening due to the irradiation-induced formation of dislocation loops and precipitates, the A-21 steel might have an advantage. The high density of precipitate particles and dislocations associated with the precipitates could act as dominant recombination sites for the vacancies and interstitials formed during irradiation, thus retarding the irradiation hardening that causes embrittlement. The particles could also trap helium, thus ameliorating any effect it might have

Table 1
Charpy properties of steels

Steel	Transition temperature[a] (°C)	Upper-shelf energy (J)
A-21	−59	13.7
9Cr–1MoVNb (T91)[b]	−64	10.5
12Cr–1MoVW (HT9)[c]	−35	7.6
ORNL 9Cr–2WVTa[d]	−94	10.8
F82H[e]	−82	10.8

[a] Transition temperature was determined midway between the upper and lower shelf energies.
[b] Heat treatment for 9Cr–1MoVNb: austenitized 1 h at 1040°C, AC; tempered 1 h at 760°C.
[c] Heat treatment for 12Cr–1MoVW: austenitized 1 h at 1040°C, AC; tempered 2.5 h at 780°C.
[d] Heat treatment for 9Cr–2WVTa: austenitized 0.5 h at 1050°C, GC; tempered 1 h at 750°C.
[e] Heat treatment for F82H: austenitized 1 h at 1040°C, AC; tempered 1 h at 750°C.

on embrittlement. This needs to be verified by irradiation experiments.

Crack initiation in steels occurs at precipitate particles, and in the conventional and reduced-activation steels, initiation probably occurs at large $M_{23}C_6$ particles, which are the dominant precipitates in these steels. If these large precipitates could be avoided, the impact toughness should be improved. In the thermomechanical treatment of the A-21 used to produce the TiC, the objective is to use up the carbon to form TiC, thus avoiding the formation of $M_{23}C_6$. Based on the TEM, this has occurred. Due to the smaller precipitates in the A-21, crack initiation must occur at a higher stress than in the 9Cr–1MoVNb. Another advantage of the A-21 for the impact properties is that it has a smaller prior-austenite grain size than the other steels.

If the operating temperature of a fusion system with a ferritic/martensitic steel is to be increased, the creep strength of the steel must be improved over that of conventional or reduced-activation steels. Although no creep tests were conducted in this work, tests have been conducted previously [9]. In Fig. 3, a comparison of Larson–Miller curves shows the creep-rupture behavior of A-21 to be superior to that of modified 9Cr–1Mo steel. A similar result is expected for the HT9 and the reduced-activation steels. This is expected, given the microstructure of A-21. Most of the Larson–Miller data for the A-21 was obtained for steel tempered at 700°C, the same heat treatment used in the present work. However, four of the points on the curve were for untempered specimens. As indicated in Fig. 3, it appears that results for all of the specimens – tempered or untempered – fall on the same smooth curve. If the steel could be used without a temper, that would be a further advantage for the steel. Use without tempering appears possible, since the carbon is incorporated in the TiC precipitate during hot working, which means a low-carbon, and thus softer, martensite results.

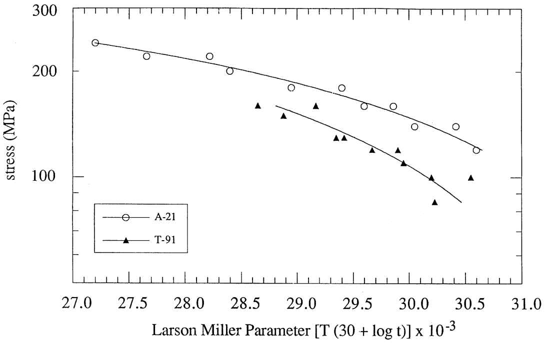

Fig. 3. A comparison of the Larson–Miller curves of A-21 and modified 9Cr–1Mo steel.

The A-21 offers another advantage. If no $M_{23}C_6$ forms, essentially all the chromium remains in solution, thus enhancing the elevated-temperature oxidation and corrosion resistance. Over 1.5% of the 9% Cr in a conventional steel can be lost from the matrix by precipitation [10].

5. Summary and conclusion

By hot working the A-21 steel in the austenitic condition, a high number density of fine TiC particles are produced on dislocations generated during the hot working. No large grain boundary and matrix $M_{23}C_6$ precipitates of the type found in conventional Cr–Mo and reduced-activation Cr–W steels were observed. Below about 600°C, the strength of the A-21 steel is lower than that of the modified 9Cr–1Mo, Sandvik HT9, F82H, and ORNL 9Cr–2WVTa, but it becomes stronger at higher temperatures. The Charpy impact properties for A-21 were similar to those of conventional Cr–Mo steels and reduced-activation Cr–W steels. The A-21 steel has superior creep properties to the modified 9Cr–1Mo and other conventional or reduced-activation ferritic/martensitic steels. All indications are that the properties of A-21 steel should allow for a significantly higher operating temperature of a fusion power plant if the first wall were constructed of A-21 instead of a conventional Cr–Mo or a reduced-activation Cr–W steel.

References

[1] R.F. Buck, Adv. Mater. Processes 150 (8) (1996) 27.

[2] D.J. Alexander, R.K. Nanstad, W.R. Corwin, J.T. Hutton, in: A.A. Braun, N.E. Ashbaugh, F.M. Smith (Eds.), Automation Technology to Fatigue and Fracture Testing, ASTM STP 1092, American Society for Testing and Materials, Philadelphia, 1990, p. 83.

[3] W.R. Corwin, A.M. Hougland, in: W.R. Corwin, G.E. Lucas (Eds.), The Use of Small-scale Specimens for Testing Irradiated Material, ASTM STP 888, American Society for Testing and Materials, Philadelphia, 1986, p. 325.

[4] D.J. Alexander, R.L. Klueh, in: J.M. Molt (Ed.), Charpy Impact Test: Factors and Variables, ASTM STP 1072, American Society for Testing and Materials, Philadelphia, 1990, p. 179.

[5] M.A. Sokolov, R.K. Nanstad, in: D.S. Gelles, R.K. Nanstad, A.S. Kumar, E.A. Little (Eds.), Effects of Radiation on Materials, Proceedings of the 17th International Symposium, ASTM STP 1270, American Society for Testing and Materials, Philadelphia, 1996, p. 384.

[6] R.L. Klueh, D.J. Alexander, in: A.S. Kumer, D.S. Gelles, R.K. Nanstad, E.A. Little (Eds.), Effects of Irradiation on Materials, Proceedings of the 16th International Symposium, ASTM STP 1175, American Society for Testing and Materials, Philadelphia, 1993, p. 591.

[7] R.L. Klueh, M.A. Sokolov, K. Shiba, Y. Miwa, J.P. Robertson, these Proceedings, p. 478.

[8] J.R. DiStefano, V.K. Sikka, Summary of modified 9Cr–1Mo steel development program: 1975–1985, ORNL-6303, Oak Ridge National Laboratory, October 1986.

[9] R.F. Buck, unpublished research, 1996.

[10] R. Jayaram, R.L. Klueh, Met. Mater. Trans. A 29A (1998) 1551.