

## **A POTENTIAL FERRITIC/MARTENSITIC STEEL FOR FUSION APPLICATIONS—R. L. Klueh, N. Hashimoto (Oak Ridge National Laboratory), R. F. Buck (Advanced Steel Technology, and M. A. Sokolov (Oak Ridge National Laboratory)**

### **OBJECTIVE**

This work was conducted as part of the effort to develop a ferritic/martensitic steel for fusion applications at higher temperatures than the conventional or reduced-activation steels are capable of operating at.

### **SUMMARY**

The A-21 steel is an Fe-Cr-Co-Ni-Mo-Ti-C steel that is strengthened by a fine distribution of small titanium carbide (TiC) precipitates formed by thermo-mechanical treatment. After a high-temperature austenitization treatment, the steel is cooled to an intermediate temperature and hot worked in the austenitic condition. During hot working, small TiC precipitates form on the dislocations generated by the working. When cooled to ambient temperature, martensite forms; finally, the steel is tempered. Transmission electron microscopy of the A-21 reveals a high number density of small TiC particles uniformly distributed in the matrix. The strength of the A-21 is less than the average value for modified 9Cr-1Mo below 600°C, but is greater above 600°C. In a Charpy impact test, the transition temperature of A-21 is similar to that of modified 9Cr-1Mo, but the upper-shelf energy is higher. Because of 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.

### **PROGRESS AND STATUS**

#### **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), that are being considered for a fusion power plant 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.

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

Numerous attempts have been made to extend the upper operating temperature for the conventional ferritic/martensitic steels. Early modifications of the Cr-Mo steels led to modified 9Cr-1Mo or Sandvik HT9, which have been considered for fusion applications. In recent years, the conventional Cr-Mo steels have been further modified by replacing some or all of the molybdenum in the composition by tungsten [1]. With these efforts, operating temperatures have been pushed to a maximum of  $\approx 620^\circ\text{C}$ .

Dispersion strengthening is the most likely mechanism available to provide the creep strength required for higher operating temperatures for ferritic/martensitic steels. The low number density of relatively large precipitates in conventional or reduced-activation ferritic/martensitic steels is not

capable of providing the strength required for temperatures beyond  $\approx 600^\circ\text{C}$ . This has led to the proposal to use ODS steels.

Obviously, a ferritic or martensitic steel that could be used at temperatures to  $650^\circ\text{C}$  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 [2]. Although this steel might not be directly applicable for fusion, the technique used to develop this steel may be applicable to produce an acceptable steel.

In this paper, tensile and impact properties have been determined for a heat of A-21 steel, and the results have been compared with properties for modified 9Cr-1Mo (9Cr-1MoVNb) and Sandvik HT9 (12Cr-1MoVW).

### 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. %) [2]. The 181-kg heat of steel used for this study was produced as a 17.5-mm thick plate that was austenitized at  $>1100^\circ\text{C}$  to dissolve the carbides and put all elements into solution. Austenitization was followed by cooling to an intermediate temperature ( $700\text{--}1000^\circ\text{C}$ ), where the steel was hot worked in the austenitic condition. After the hot-working procedure was complete, the steel was cooled to ambient temperature to transform the matrix to martensite. Finally, the steel was tempered in the range  $650\text{--}750^\circ\text{C}$  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 were machined from the tempered plate with gage lengths parallel to the rolling direction. Tensile tests were conducted over the range room temperature to  $700^\circ\text{C}$  in vacuum on a 44-kN Instron universal testing machine at a nominal strain rate of  $\approx 4 \times 10^{-4} \text{ s}^{-1}$ .

One-third-size Charpy specimens  $3.3 \times 3.3 \times 25.4$  mm with a 0.51-mm-deep  $30^\circ$  V-notch and a 0.05- to 0.08-mm-root radius were machined from the plate along the rolling direction with the notch transverse to the rolling direction. Charpy tests were carried out in a pendulum-type impact machine specially modified to accommodate subsize specimens [3]. The absorbed energy values were fitted with a hyperbolic tangent function to permit the upper-shelf energy (USE) and ductile-brittle transition temperature (DBTT) to be evaluated. The DBTT was determined at the energy midway between the upper- and lower-shelf energies. Note that for these miniature specimens different DBTT and USE values are obtained than for full-size specimens. However, it has been shown that a low transition temperature for miniature specimens translates to a low value for full-size specimens [4-6]. A correlation likewise exists for the USE [4-6].

Properties of A-21 were compared against those of modified 9Cr-1Mo (9Cr-1MoVNb—T91) steel, a conventional Fe-9Cr-1Mo-0.2V-0.07Nb-0.03N-0.1C steel and Sandvik HT9 (12Cr-1MoVW), a conventional Fe-12Cr-1Mo-0.5W-0.5Ni-0.25V-0.2C steel [7]. Both of these steels were once considered for fusion applications. These steels were tested in their standard heat treated conditions. For the 9Cr-1MoVNb, austenitization was at  $1040^\circ\text{C}$  with tempering 1 h at  $760^\circ\text{C}$ , and for the HT9 austenitization was at  $1040^\circ\text{C}$  followed by a 2.5 h temper at  $780^\circ\text{C}$ .

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 ( $\text{LaB}_6$ ) microscope. Foil thicknesses were measured by thickness fringes in order to quantitatively determine the number density of the precipitates.

## Results

### Microstructure

Observation by optical microscopy indicated the steel had a 100% tempered martensite structure with a prior austenite grain size of 5-15 $\mu\text{m}$  [Fig. 1(a)]. The TEM examination at low magnification revealed a subgrain structure typical of tempered martensite [Fig. 1(b)].

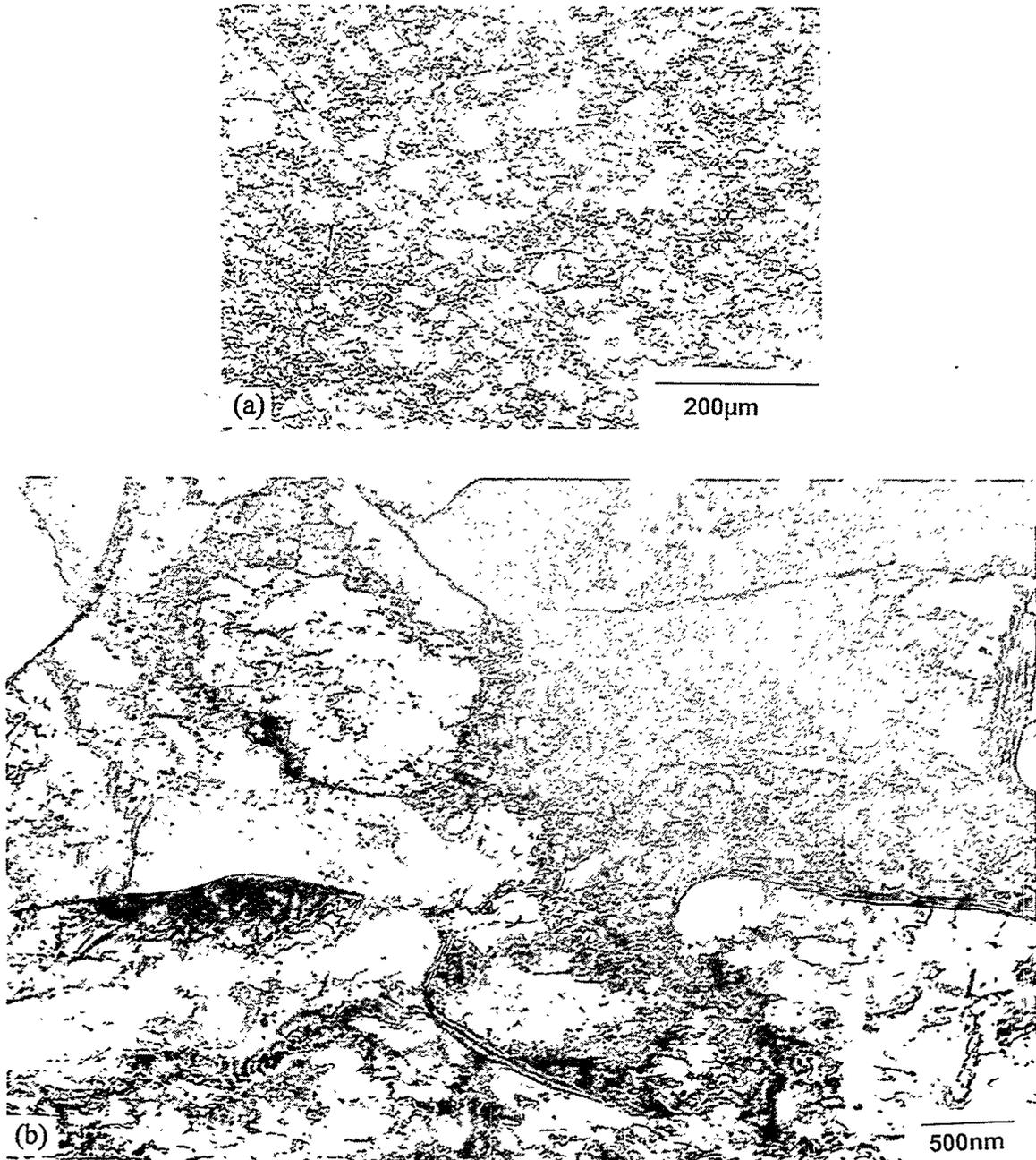


Figure 1. The (a) optical and (b) transmission electron microscopy views of the tempered martensite microstructure of A-21 steel.

The subgrains contain a high number density of precipitates [Figs. 2(a) and 2(b)] uniformly distributed with no indication of denuded zones near 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. 2(c) and 2(d)].

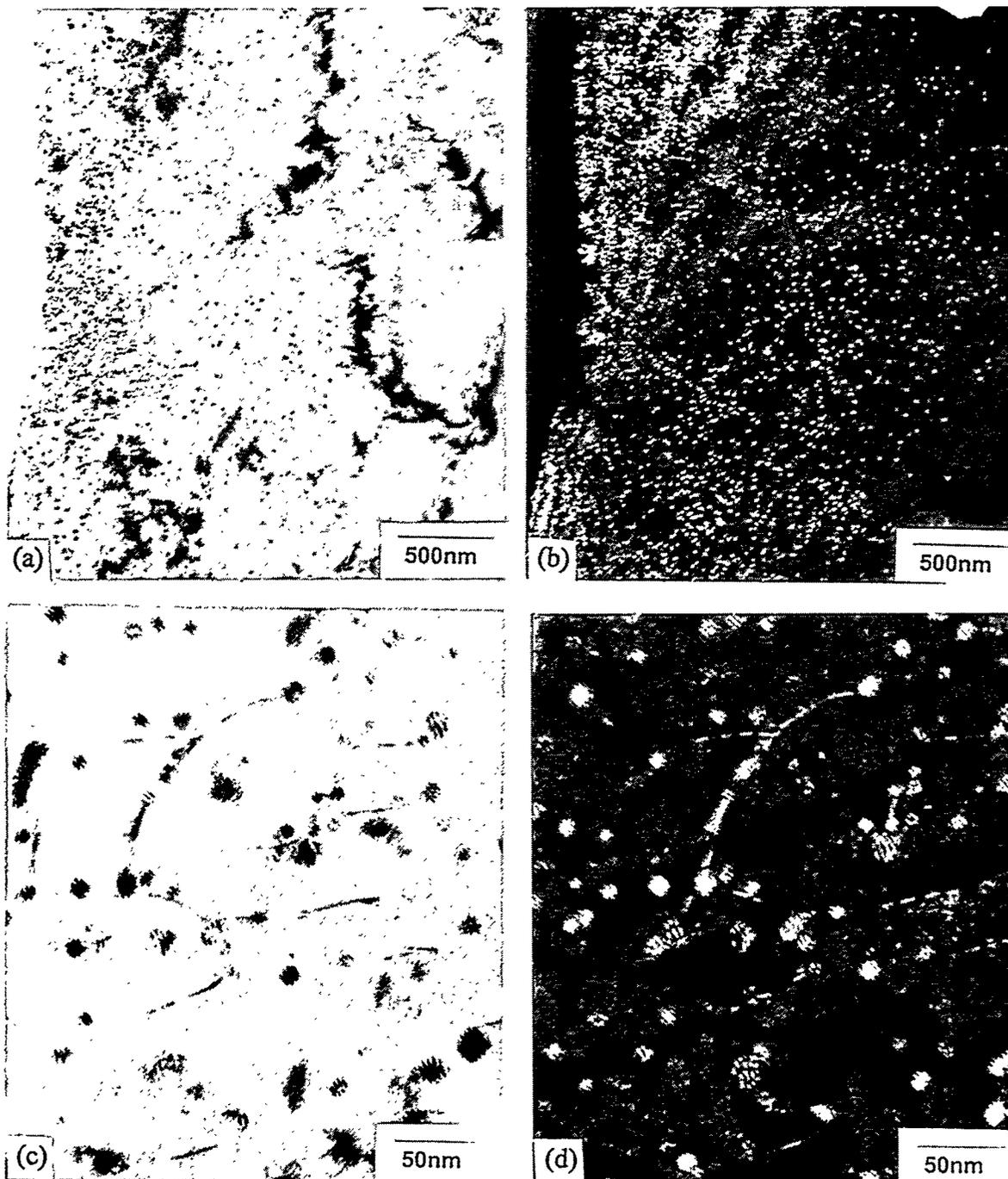


Figure 2. Transmission electron micrograph showing high number density of TiC precipitates in (a) bright field and (b) dark field and showing precipitates on dislocations in (c) bright field and (d) dark field.

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 (Fig. 3), with the average size about 9.3 nm. The total number density was estimated to be  $4.7 \times 10^{21} \text{ m}^{-3}$ .

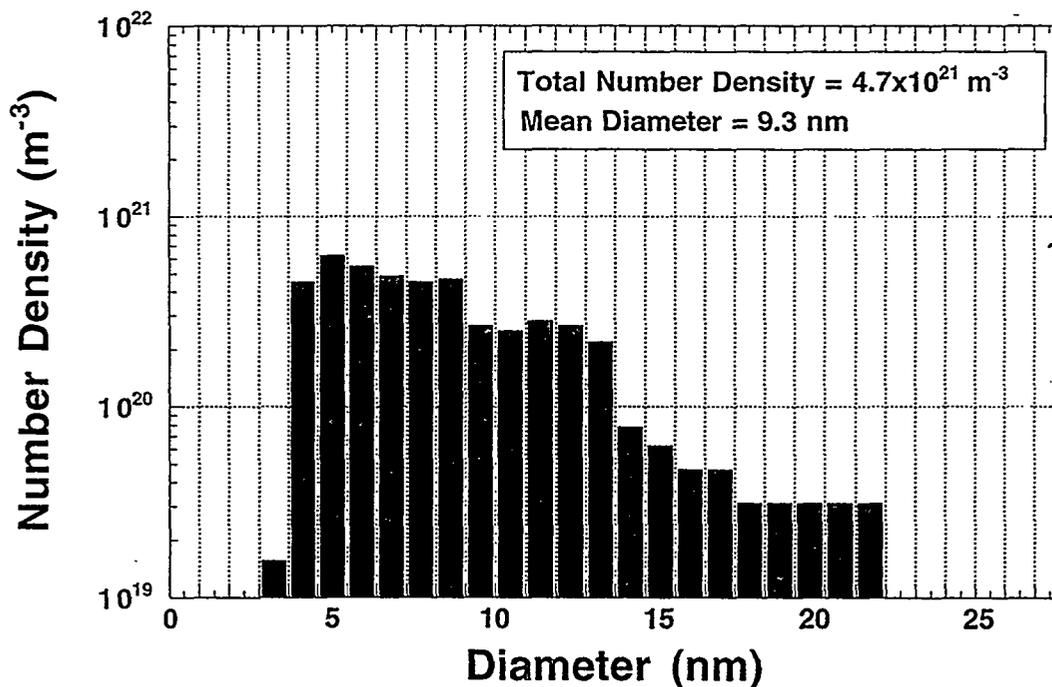


Figure 3. Size distribution of precipitates in A-21 steel.

#### Tensile Properties

Figure 4 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  $\approx 600^\circ\text{C}$  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 value for 9Cr-1MoVNb steel [8].

#### Charpy Impact Properties

The Charpy impact curve for the A-21 steel is shown in Fig. 5 compared to the curve for a heat of modified 9Cr-1Mo steel [7]. The ductile-brittle transition temperature (DBTT) for A-21 at half the upper-shelf temperature was similar to that of modified 9Cr-1Mo steel and the Sandvik HT9, while the upper-shelf energy for A-21 is significantly higher than that of both modified 9Cr-1Mo and HT9 (Table 1).

#### 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 the higher operating temperatures of a fusion

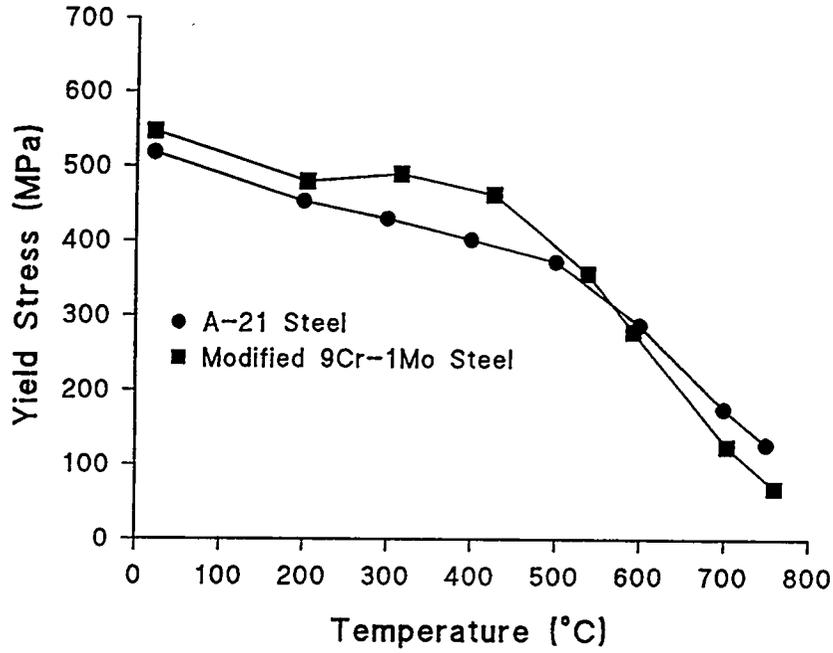


Figure 4. Tensile properties of A-21 steel compared to modified 9Cr-1Mo steel.

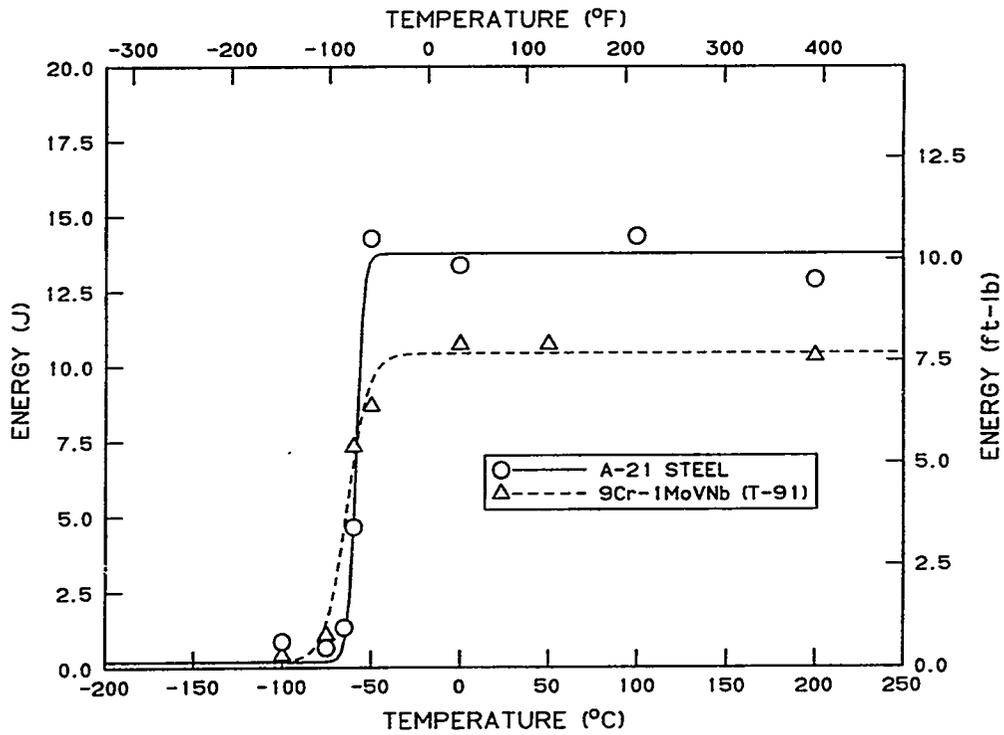


Figure 5. Charpy curves for A-21 and modified 9Cr-1Mo steels.

Table 1. Charpy Properties of Steels

Steel	Transition Temperature (°C)	Upper-Shelf Energy (J)
A-21	-59	13.7
9Cr-1MoVNb (T91) <sup>a</sup>	-64	10.5
12Cr-1MoVW (HT9) <sup>b</sup>	-35	7.6

<sup>a</sup> Heat treatment for 9Cr-1MoVNb: austenitized 1 h at 1040°C, AC; tempered 1 h at 760°C.

<sup>b</sup> Heat treatment for 12Cr-1MoVW: austenitized 1 h at 1040°C, AC; tempered 2.5 h at 780°C.

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 are expensive, and given the more conventional 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 indicate that, it has the kind of properties required for a candidate for fusion applications. Irradiation embrittlement, which results in 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 for fusion applications. Generally, steels with a low DBTT before irradiation have a low value after irradiation. The modified 9Cr-1Mo steel showed a relatively small increase in DBTT when irradiated in the absence of the formation of significant amounts of helium [7]. Since the shift in DBTT is caused by irradiation hardening due to the irradiation-induced formation of dislocation loops and precipitates, 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 on embrittlement. This needs to be verified by irradiation experiments.

Crack initiation in steels generally occurs at precipitate particles, and in the 9Cr-1MoVNb and 12Cr-1MoVW steels, initiation probably occurs at the 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 thermo-mechanical treatment of the A-21 used to produce the TiC, the objective is to use up all the carbon to form TiC, thus avoiding the formation of the  $M_{23}C_6$ . Based on the TEM, this has occurred. Because of the smaller precipitates in A-21, crack initiation must occur at a higher stress than in the 9Cr-1MoVNb. Another advantage of the A-21 for the impact tests is that it has a smaller prior-austenite grain size than the 9Cr-1MoVNb (estimated prior-austenite grain sizes were 5-15 $\mu$ m for the A-21 vs. 16-22 for the 9Cr-1MoVNb [7]).

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, such tests have been conducted previously [9]. In Fig. 6, a comparison of Larson-Miller curves shows the creep-rupture behavior of A-21 to be superior to that of the modified 9Cr-1Mo steel. This is expected, given the

microstructure. Most of the points on the Larson-Miller curve for the A-21 steel were obtained for steel that had been tempered at 700°C, the same heat treatment used in the present work. However, four of the points on the curve were for specimens without the temper. As indicated in Fig. 6, 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 essentially all carbon is incorporated in the TiC precipitate, which means a low-carbon, and thus, softer martensite results.

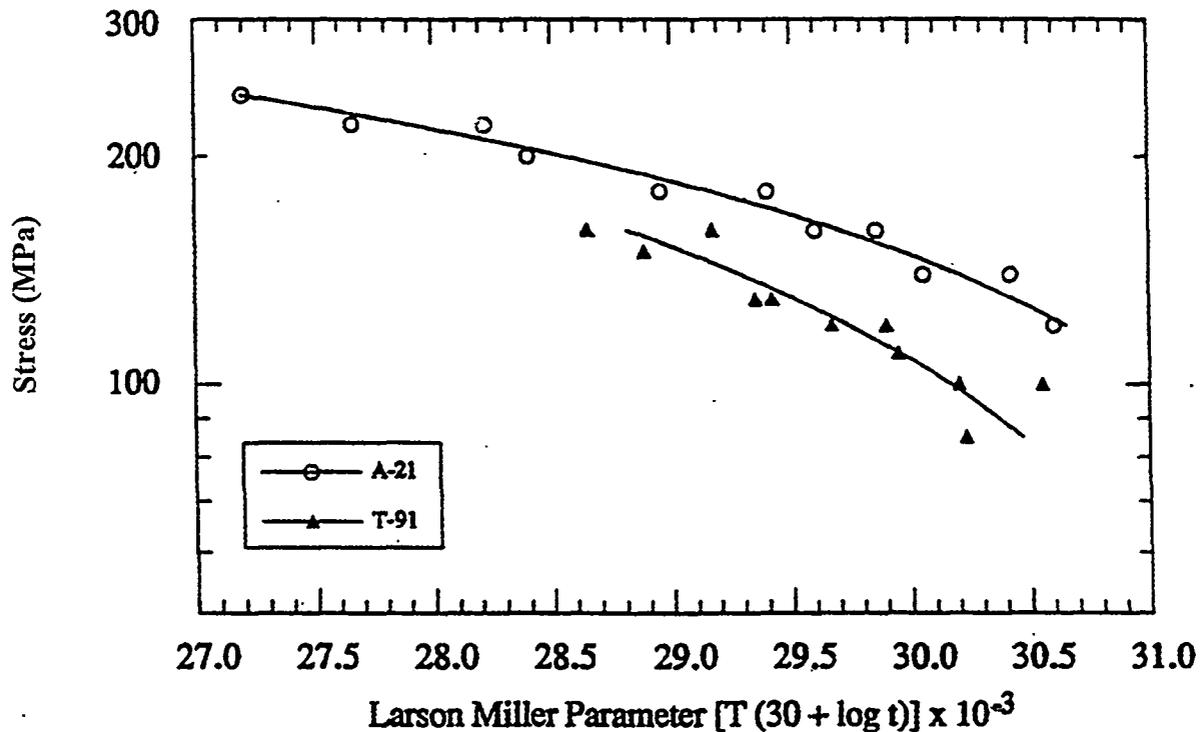


Figure 6. A comparison of the Larson-Miller curves of A-21 and modified 9Cr-1Mo steel.

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].

#### SUMMARY AND CONCLUSION

Tensile and Charpy impact properties were determined for A-21 steel, an Fe-9.5Cr-3Co-1Ni-0.6Mo-0.3Ti-0.07C ferritic/martensitic steel. Microstructure was also examined. By hot working the steel in the austenitic condition following austenitization to put all elements in solution, 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 the conventional Cr-Mo and reduced-activation Cr-W steels were observed. The strength of the A-21 steel is lower than that of modified 9Cr-1Mo steel at  $\leq 600^\circ\text{C}$ , but it becomes stronger at higher

temperatures. The transition temperature in a Charpy impact test for A-21 was similar to that of the modified 9Cr-1Mo steel, but the upper-shelf energy was higher.

Because of the presence of the high number density of fine TiC particles, 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 the 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.

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