

Available online at www.sciencedirect.com



journal of nuclear materials

Journal of Nuclear Materials 367-370 (2007) 117-121

www.elsevier.com/locate/jnucmat

Mechanical properties and microstructures of China low activation martensitic steel compared with JLF-1

Y. Li^{a,*}, Q. Huang^a, Y. Wu^a, T. Nagasaka^b, T. Muroga^b

^a Institute of Plasma Physics, Chinese Academy of Sciences, P.O. Box 1126, Hefei, Anhui 230031, China ^b National Institute for Fusion Science, Oroshi, Toki, Gifu 509-5292, Japan

Abstract

The tensile and impact properties of CLAM steel are compared to those of JLF-1 steel. Tensile testing revealed that the ultimate and yield strengths of the CLAM steel are 670 MPa and 512 MPa at room temperature, and 373 MPa and 327 MPa at 873 K, respectively. These values are higher than those measured for JLF-1. The ductile-to-brittle transition temperature (DBTT) of CLAM was found to be 171 K using one-third size Charpy V-notch specimens, which is 16 K lower than that of JLF-1. Microstructural analysis by SEM and TEM indicated that the prior austenite grain size and lath width for CLAM are smaller than those for JLF-1. The finer grain and lath structure is considered to be one of the main reasons for the higher strength and lower DBTT of the CLAM steel.

© 2007 Elsevier B.V. All rights reserved.

1. Introduction

Reduced activation ferritic/martensitic (RAFM) steels are one of the candidate structural materials for the DEMO fusion reactor because of their better swelling resistance, thermo-physical and thermo-mechanical properties, as compared to austenitic stainless steels [1]. Research on RAFM steels has been carried out in Europe, Japan and USA in the past 20 years and some inspiring progress has been made, including the development of F82H, JLF-1, and EUROFER97 steels [2].

E-mail address: yfli@ipp.ac.cn (Y. Li).

In China, the China low activation martensitic (CLAM) steel has also been developed and a series of R&D activities on CLAM are being carried out in Institute of Plasma Physics, Chinese Academy of Sciences (ASIPP) in collaboration with other institutes and universities in China and in other countries [3]. The chemical composition of the CLAM steel is based on the nominal composition 9Cr1.5WVTa. The elements Nb and Mo cause long term activation and are replaced by W, V and Ta as compared to the common martensitic steels. The Ta content is set to 0.15% to improve the properties at high temperature. The impurity elements, such as O, N, S and Nb, etc., are reduced to as low a level as possible.

In this paper, the impact and tensile properties of unirradiated CLAM steel (FDS-HEAT 0408B) are

^{*} Corresponding author. Tel.: +86 551 5592424; fax: +86 551 5593328.

^{0022-3115/\$ -} see front matter © 2007 Elsevier B.V. All rights reserved. doi:10.1016/j.jnucmat.2007.03.012

reported and compared to those of the JLF-1 steel (JOYO-2-HEAT).

2. Experimental procedure

The materials used were the CLAM (FDS-HEAT 0408B) and JLF-1 (JOYO-2-HEAT) RAFM steels. Their chemical composition is listed in Table 1. The CLAM steel was melted in a vacuum induction furnace into an ingot of 20 kg, and then it was hot-forged and rolled into a 12-mm-thick plate, while the JLF-1 steel was melted into a 100 kg ingot and fabricated as a 25-mm-thick plate. The detailed heat treatment conditions for both steels are given in Table 2.

Charpy specimens were one-third size V-notch specimens measuring $3.3 \times 3.3 \times 25.4 \text{ mm}^3$ with a 0.66-mm-deep 30° angle-V-notch and a 0.08-mmroot radius. Specimens were aligned parallel to the rolling direction. Charpy tests were conducted in the temperature range from room temperature (RT) to 123 K using a drop-tower-type impact machine. Temperature control of the specimens was performed by using a conditioning chamber where low temperatures were reached by using liquid nitrogen and cooled isopentane. The specimens were immersed in the chamber at the test temperature for about 10 min prior to testing to ensure temperature stabilization to within 2 K. The impact velocity was 4.75 m/s and the absorbed energy was electronically integrated from the load-displacement curve.

The gauge size of the tensile specimens was $5 \times 1.2 \times 0.25$ mm³. Tensile tests were conducted at RT, 673, 773 and 873 K at an initial strain rate of 6.67×10^{-4} s⁻¹. The RT test was conducted in air, while the tests at elevated temperatures were carried out in a vacuum of 10^{-3} Pa. The 0.2% proof strength was measured as yield strength.

Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) observations used a JEOL-5600 operated at 20 kV and a JEOL-2010S operated at 200 kV. The distribution of precipitates was observed in SEM to determine the prior austenite grain size after electro-etching in a $HCl-C_2H_5OH$ solution. TEM samples were electro-

Table 1 Chemical compositions of CLAM and JLF-1 steels in wt%

Table	2
raute	_

Heat	treatment	conditions	for	CLAM	and JLF-1	steels
		••••••••••		~ ~ ~ ~ ~ ~ ~		

Type of steel	Normalization	Tempering
CLAM	1253 K/30 min/air cool	1033 K/90 min/air cool
JLF-1	1323 K/60 min/air cool	1053 K/60 min/air cool

lytically thinned using a solution of $CH_3COOH-HClO_4$ and the microstructure was observed.

3. Results and analysis

3.1. Tensile properties

The tensile specimens were tested from RT to 873 K with steps of 100 K. The results for CLAM and JLF-1 steels are shown in Fig. 1. It can be seen that the strength of the CLAM steel is higher than that of the JLF-1 steel at all tested temperatures. For the CLAM steel, the ultimate tensile strength (UTS) is 670 MPa and the yield strength (YS) is 512 MPa at RT, while they are equal to 373 MPa and 327 MPa at 873 K, respectively. The differences in UTS and YS between the CLAM and JLF-1 steels are 32 MPa and 35 MPa at RT, and 30 MPa and 27 MPa at 873 K, respectively. The differences are a little larger at 673 K and 773 K. The total elongations show no major difference and are thought to be similar for the two steels at all test temperatures.

3.2. Impact properties

The impact properties of the CLAM and JLF-1 steels are shown in Fig. 2.

The tests were performed in the temperature range 123 K to RT. Charpy data were fitted with a hyperbolic tangent function for obtaining the transition temperature. The DBTT reported here was defined as the temperature corresponding to half the difference between the upper-shelf energy (USE) and lower-shelf energy (LSE), which is different from the definition where the DBTT is the temperature corresponding to 50% of the USE [4].

	Cr	W	С	Mn	V	Та	0	Ν	Р	S	Fe
CLAM	8.91	1.44	0.12	0.49	0.20	0.15	_	_	_	_	Bal.
JLF-1	9.00	1.98	0.09	0.49	0.20	0.083	0.0019	0.0150	< 0.003	0.0005	Bal.



Fig. 1. Tensile properties: (a) UTS, YS and (b) total elongation of CLAM and JLF-1 steels.



Fig. 2. Impact energy-temperature curves for CLAM and JLF-1 steels.

The DBTT of the CLAM steel is about 171 K, which is 16 K lower than that of the JLF-1 steel. The USE of CLAM is slightly lower than that of JLF-1.

3.3. Microstructure

SEM observations of surfaces of the steels are shown in Fig. 3. In these images, since the precipitates exhibit a strong bright contrast, allow the prior austenitic grain boundaries to be seen. The prior austenite grain size in the case of the CLAM steel is significantly smaller than in the JLF-1 steel.

TEM images are shown in Fig. 4. It can be seen that the microstructure of both steels consists of a mixture of lath-martensite phase and well-tempered martensite phase. In addition, there is less lath-martensite near the center of the plate than at the surface for the CLAM.

The lath width was determined by counting the number of intersections of lath boundaries with straight lines vertical to elongated direction of lath grains. The lath width in the CLAM steel was smaller than that in the of JLF-1 steel.

There is no difference in the average size of carbides in the two steels.



Fig. 3. SEM images showing the distribution of precipitates in (a) CLAM and (b) JLF-1 steels.



Fig. 4. TEM images taken near: (a) the surface of the CLAM plate; (b) the center of the CLAM plate; and (c) the center of the JLF-1 plate.

Table 3 Parameters characterizing the microstructure of CLAM and JLF-1 steels

	CLAM (µm)	JLF-1	
Prior austenite grain size	5.5	9.9	
Lath width	0.30	0.50	
Carbide size	0.104	0.104	

Table 3 summarizes the parameters describing the microstructure of CLAM and JLF-1 steels.

4. Discussion

As can be seen from the comparison of the data in Figs. 1 and 2, the strength of the CLAM steel is higher and its DBTT is lower than those of JLF-1. The main microstructural differences in the two steels relate to the prior austenite grain size and the lath width, which could account for the difference in Charpy impact DBTT and tensile strength in the normalized-and-tempered condition.

It is well known that the effect of grain size on the yield strength is described by the Hall–Petch's equation [5]:

$$\Delta \sigma_{\rm y} = \Delta (k_{\rm b} D^{-1/2}), \tag{1}$$

where σ_y is the yield strength, k_b a constant related to be interaction between grain boundaries and dislocations, and D is the grain diameter.

In general, a finer grain diameter gives a better yield strength and toughness. This effect could be estimated roughly by using the above equation.

Because RAFM steels have anisotropic and very fine lath structure, it is difficult to determine the effective D. In Ref. [6], the packet diameter was thought to be more acceptable for D than the prior austenite grain diameter. The packet boundaries

consist of both prior austenite grain boundaries and lath boundaries. In Ref. [7], the average lath diameter was used as the effective D in the case of the lath structure by considering the extent of the slip planes in the lath. Because there is not 100% lath-martensite in the CLAM and JLF-1 steels, the packet diameter and length were hard to determine, and thus the prior austenite grain diameter was used in this study to estimate the difference in YS between the CLAM and JLF-1 steels.

The grain size in the CLAM steel is about 5.5 μ m, which was almost half the value in the JLF-1 steel. The factor $k_{\rm b}$ was determined to be 0.62 M Nm^{-1.5} according to Ref. [8], so the difference in YS between CLAM and JLF-1, $\Delta \sigma_{\rm y}$, was calculated to be about 67 MPa.

The experimental difference in YS between the CLAM and JLF-1 steels is 35 MPa at RT, as shown in Fig. 1, which is smaller than the calculated value. The possible reason for this could be due to the smaller content of W in the CLAM than in the JLF-1 steel, because W should contribute to the strength by solid solution strengthening [9].

One of the reasons for finer prior austenite grain size and lath width in the CLAM steel is the heat treatment. Some earlier studies have shown [10,11] that mechanical properties can be modified by variations of the normalization (quenching) temperature and the tempering temperature. In the present case, the normalization was performed at 1253 K for 30 min for CLAM, and at 1323 K for 60 min for JLF-1. Lower normalization temperature and shorter time for CLAM lead to finer prior austenite grains and to a slight decrease of the DBTT. In addition, the tempering temperature was 1033 K for CLAM and 1053 K for JLF-1. The lower tempering temperature produces a smaller lath width and then a slight decrease of the DBTT. The CLAM steel contains 0.15 wt% Ta, which is higher than in the JLF-1 steel (0.083 wt% Ta). The higher content of Ta is another possible reason for the finer prior austenite grain size and lath width. It has been reported that Ta has a beneficial effect on both DBTT and strength [12,13]. In the 9%Cr– 2%W steel, the addition of 0.05%Ta resulted in finer grain sizes for all austenization temperatures from 1223 K to 1423 K, and caused an increase in the strength at elevated temperatures and a decrease of the toughness at low temperatures [12]. The positive effect of Ta was also observed in 5Cr and other 9Cr steels [13]. Ta, like Nb, is a strong carbide former, and can retard the growth of austenite in heating process and inhibit austenite grain growth.

In addition, Klueh et al. [14] offered an explanation for the beneficial effects of Ta on the impact properties of Ta-bearing steels. It seems that sufficient Ta available in solid solution increases the cleavage stress or affects the temperature relationship of the flow stress advantageously. It has been also observed that the impact properties are degraded with increasing irradiation dose and decreasing irradiation temperature, indicating that Ta might be lost from solution by precipitation during irradiation. However, the mechanism of Ta effect is not yet been fully understood.

5. Conclusion

The impact and tensile properties of the CLAM (FDS-HEAT 0408B) steel were studied and compared to those of the JLF-1 (JOYO-2-HEAT) steel. The following main conclusions were obtained:

- (1) The DBTT of the CLAM steel is 171 K, which is 16 K lower than that of the JLF-1 steel.
- (2) The strength of the CLAM steel is higher than that of the JLF-1 steel at all tested temperatures.
- (3) The prior austenite grain size in the CLAM steel is only half the size of that in the JLF-1 steel, and the width of martensite lath is smaller than that in JLF-1.

- (4) The mean size of carbides is almost the same in the CLAM and JLF-1 steels.
- (5) Lower heat treatment temperature and a higher Ta content are considered as the main reasons leading to reduced prior austenite grain size and martensite lath width in the CLAM steel. The finer grain and lath structure is one of the reasons for the higher strength and lower DBTT of the CLAM steel.

Acknowledgements

This work was supported by the National Natural Science Foundation (Grant No. 10375067), the JSPS-CAS Core University Program, NIFS Budget Code NIFS 05UCFF005 and the Knowledge Innovation Program of Chinese Academy of Sciences.

References

- T. Muroga, M. Gasparotto, S.J. Zinkle, Fus. Eng. Des. 61&62 (2002) 13.
- [2] S. Jitsukawa, A. Kimura, A. Kohyama, R.L. Klueh, J. Nucl. Mater 329–333 (2004) 39.
- [3] Q.Y. Huang, J.N. Yu, F.R. Wan, et al., J. Nucl. Sci. Eng. 24 (3) (2004) 56 (in Chinese).
- [4] H. Kayano, H. Kurishita, A. Kimura, et al., J. Nucl. Mater. 179–181 (1991) 425.
- [5] N.J. Petch, J. Iron Steel. Inst. 173 (1953) 25.
- [6] T. Nagasaka, Hiroo Yoshida, Ken-ichi Fukumoto, et al., Mater.Trans. JIM 41 (1) (2000) 170.
- [7] J.P. Naylor, Metall. Trans. 10 (A) (1979) 861.
- [8] M. Klesnil, M. Holzmann, P. Lukas, P. Rys, J. Iron Steel. Inst. (1965) 47.
- [9] S.G. Hong, W.B. Lee, C.G. Park, J. Nucl. Mater. 288 (2001) 202.
- [10] R.L. Klueh, D.J. Alexander, J. Nucl. Mater. 265 (1999) 262.
- [11] L. Schafer, M. Schirra, J. Nucl. Mater. 271&272 (1999) 455.
- [12] H. Kayano, A. Kimura, M. Narui, et al., J. Nucl. Mater. 155–157 (1988) 978.
- [13] R.L. Klueh, D.J. Alexander, M.A. Sokolov, J. Nucl. Mater. 304 (2002) 139.
- [14] R.L. Kluch, D.J. Alexander, M. Rieth, J. Nucl. Mater. 273 (1999) 146.