



R&D of oxide dispersion strengthened ferritic martensitic steels for FBR

S. Ukai ^{a,*}, T. Nishida ^{a,b,1}, T. Okuda ^b, T. Yoshitake ^a

^a O-arai Engineering Center, Power Reactor and Nuclear Fuel Development Corporation, 4002, Narita, O-arai, Ibaraki 311-1393, Japan

^b Kobe Steel Ltd., Kobe, Hyogo, Japan

Abstract

As prospective cladding material for the long-life core of a Fast Breeder Reactor (FBR), we developed oxide dispersion strengthened (ODS) ferritic/martensitic steels, which have more swelling resistance than austenitic steels and are expected to have a superior creep strength at elevated temperatures. In order to improve the inferior strength in the hoop direction of manufactured ODS cladding tubes, recrystallization and martensitic phase transformation techniques have been developed, and the strength anisotropy was successfully improved in laboratory scale tests. It is also demonstrated that cold rolling manufacturing for the ODS ferritic cladding was possible using the recrystallization technique. © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

The long life core materials used in a Fast Breeder Reactor (FBR) environment are required to have superior resistance to high neutron exposure beyond 200 dpa as well as good high temperature mechanical properties at around 973 K. For prospective cladding materials for a long-life core, we considered oxide dispersion strengthened (ODS) ferritic/martensitic steels, which have more swelling resistance than austenitic steels and are expected to have superior creep strength at elevated temperature. These ODS ferritic/martensitic steels are directly applicable to fusion materials, because the selected composition is of low activation.

Research and development of the ODS ferritic/martensitic steels was started in 1987. Fundamental studies concerning optimization of the mechanical alloying (MA) process as well as effects of alloying elements on high temperature mechanical strength were carried out in cooperation with fabrication vendors [1–3]. Based on the results of these studies, the manufacturing of thin-walled cladding tubes was initially attempted using hot

extrusion and warm rolling processes in 1989 [4]. These ODS cladding tubes had strong anisotropy with superior strength in the longitudinal direction, but inferior strength in the perpendicular direction to rolling. These unexpected features of ODS ferritic steels are due to formation of a bamboo grain structure excessively elongated along the rolling direction.

Based on this basic research [5], technology was developed to destroy the elongated bamboo structure and to control the grain morphology of ODS cladding tubes using both recrystallization processes [6] and phase transformation processes [7]. From 1995, using both processes, cladding tube manufacturing has been conducting using cold-rolling techniques [8]. This paper describes the R&D activity that was conducted for improving the mechanical properties of ODS ferritic/martensitic steels and for cold-rolling manufacturing of cladding tubes.

2. Recrystallized ODS ferritic steels

2.1. Recrystallization characteristics

Controlling the grain structure of the ODS ferritic steels was attempted using recrystallization processes. The oxide particles act by resisting cold-working

* Corresponding author. Tel.: +81 29 267 4141; fax: +81 29 266 3713; e-mail: uki@oec.pnc.go.jp.

¹ Present address: Kobe Steel Ltd., Kobe, Hyogo, Japan.

deformation, and retard dislocation recovery and recrystallization. Hence, recrystallization in ODS ferritic steels is controlled by the Y_2O_3 content. A parametric study with various Y_2O_3 contents was carried out at an annealing temperature of 1473 K after about 60% cold-working, and the recrystallized region was demonstrated to be restricted to Y_2O_3 contents less than 0.25% [6].

Creep rupture and ring tensile tests on the recrystallized ODS ferritic steel tubes were conducted utilizing tubes machined from recrystallized bars. As shown in Fig. 1, there is a distinct difference in longitudinal uni-axial and bi-axial hoop directions of previously manufactured 1DK cladding containing anisotropy grain structure [4]. However, strength anisotropy for uni-axial and bi-axial hoop directions completely disappears in the recrystallized ODS ferritic steel tube. From ring tensile tests, it was also demonstrated that the uniform elongation in the hoop direction of the recrystallized ODS ferritic steel tube was adequately improved beyond 5% [6].

2.2. Cladding tube manufacturing

Based on the results of laboratory scale experiments to promote recrystallization, a manufacturing study of the thin-walled cladding tubes with cold-working was extensively conducted on an industrial scale by tube

manufacturing vendors [8]. The recrystallization following cold-rolling was able to soften the hardened cladding tube and made it possible for cold-rolling at room temperature, although the previously manufactured 1DK and 1 DS cladding tubes had to be rolled at intermediate temperatures of 723–1073 K [4].

From the material viewpoint, the Y_2O_3 content should be kept below 0.25 mass%, in order to allow recrystallization at appropriate elevated temperatures following cold-rolling. The Ti content should be also decreased, because relatively large particles of Ti-oxide and/or Ti-carbo-nitride may retard recrystallization and provide a source for cracking during the cold-rolling process. The amount of cold-working level per pass and the appropriate ratio of reduction in diameter to thickness (Q -value) to reduce the residual stress across the cladding thickness were optimized from the forming point of view.

The hot extrusion process at 1423 K is based on the composition Fe–11.5Cr–2.2W–0.23Ti–0.015C–0.2 Y_2O_3 (mass%), which includes lower Ti and Y_2O_3 contents to provide better workability for cladding manufacturing. The manufactured bars were machined to 15.25 mm outer diameter and 12.00 mm inner diameter. Then, the cold-rolling was repeated 10 times with about 15% cold-worked level in each pass. A recrystallization heat treatment at 1373 K for 4 min was included after every

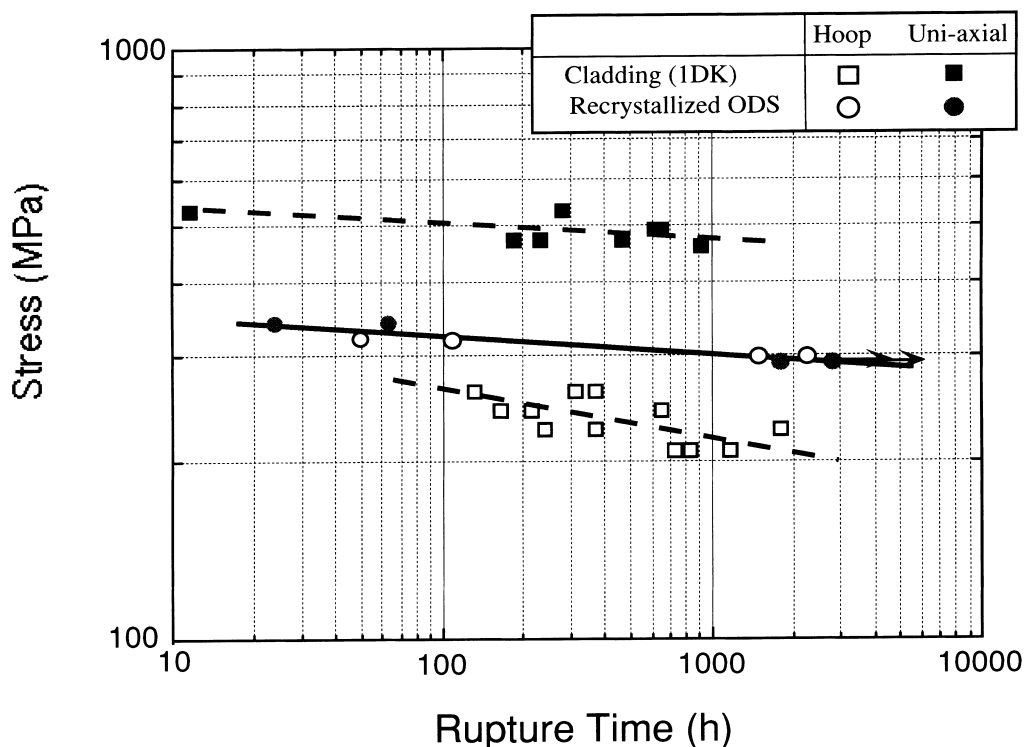


Fig. 1. Creep rupture properties of recrystallized ODS ferritic steel tube, compared with unrecrystallized cladding tube (1DK).

two cold-rolling steps, in order to soften the cold-worked cladding tubes. Finally, the plug drawing to 4% of cold work was used to reach precise final dimensions of 6.5 mm outer diameter and 0.47 mm thickness. The Q -value adopted was 1.5 for the entire rolling process, which maintains a slightly compressive stress state on the cladding outer surface. Fig. 2 shows the results of the hardness measurement at each step of the cold-working and recrystallization heat treatment process as an indicator for workability. In this figure, L and T directions represent the hardness in longitudinal and transverse sections. It is apparent that intermediate recrystallization heat treatments adequately soften the hardened cladding tube to hardness levels of around 350 Hv, which is necessary for industrial scale manufacturing of cladding tubes. No defects or cracks are found by ultrasonic testing, and sufficient ductility in the hoop direction was demonstrated in the manufactured cladding tubes.

3. ODS martensitic steels

3.1. Martensitic characteristics

In order to remove the elongated grain structure and control the grain size in previously manufactured cladding tubes, a martensitic phase transformation approach was utilized. The α to γ phase transformation can be

induced in martensitic steels with carbon in the range 0.1–0.2 mass% and chromium contents at lower of 9–11 mass%. It was demonstrated that strength anisotropy could be nearly eliminated in this 11Cr ODS martensitic steel [7].

The typical creep rupture strength of 11Cr ODS martensitic steel tubes is shown in Fig. 3, as compared with that of ferritic/martensitic steel designated PNC-FMS [9]. The internal creep rupture strength of the ODS martensitic steel with a composition of 11Cr–0.2C–2.5W–0.5Ni–0.43Ti–0.93Y₂O₃ (mass%) should be adequately improved beyond that of PNC-FMS. It is well known that the addition of Y₂O₃ alone improves strength, but the magnitude of the increase is not due only to Y₂O₃ additions. Addition of 0.43 mass% Ti to the ODS martensitic steel also increased the strength significantly due to the formation of a finely distributed Y₂O₃–TiO₂ complex oxide particles [7].

3.2. Heat treatment

The heat treatment of the ODS martensitic steels was investigated in order to make it possible to manufacture the cladding tubes. In this test, the ODS martensitic steel with composition 11Cr–0.2C–2.8W–0.42Ti–0.36Y₂O₃ (mass%) was used. The normalized condition, 1323 K for 1 h followed by air cooling (AC) to room temperature, gives hardness values as high as 510 HV as shown in Fig. 4. A tempering treatment which followed at

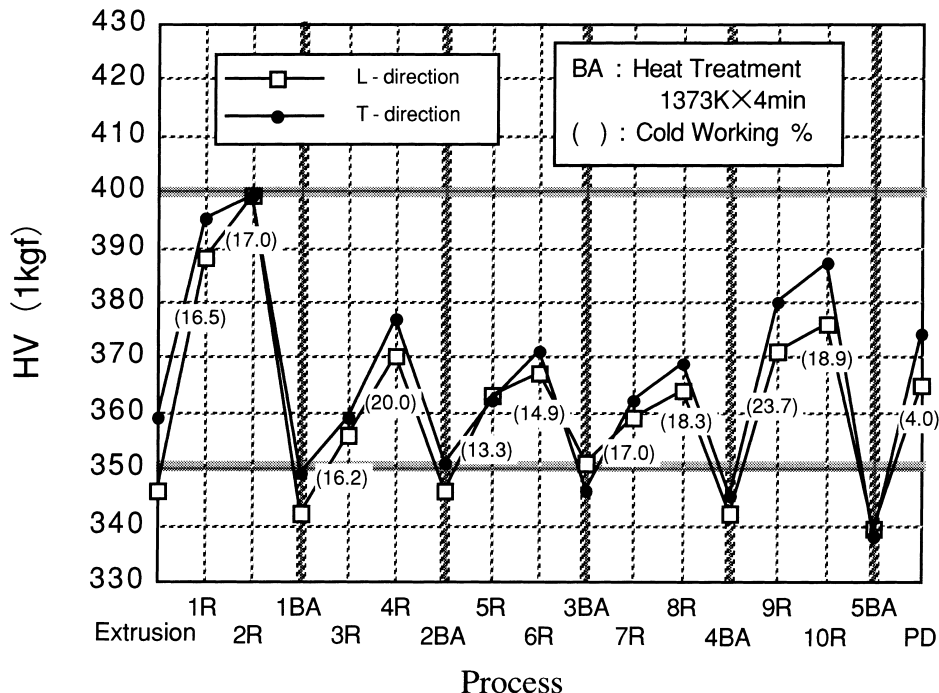


Fig. 2. Results of hardness measurement of ODS ferritic steel during cladding tube manufacturing using cold-rolling.

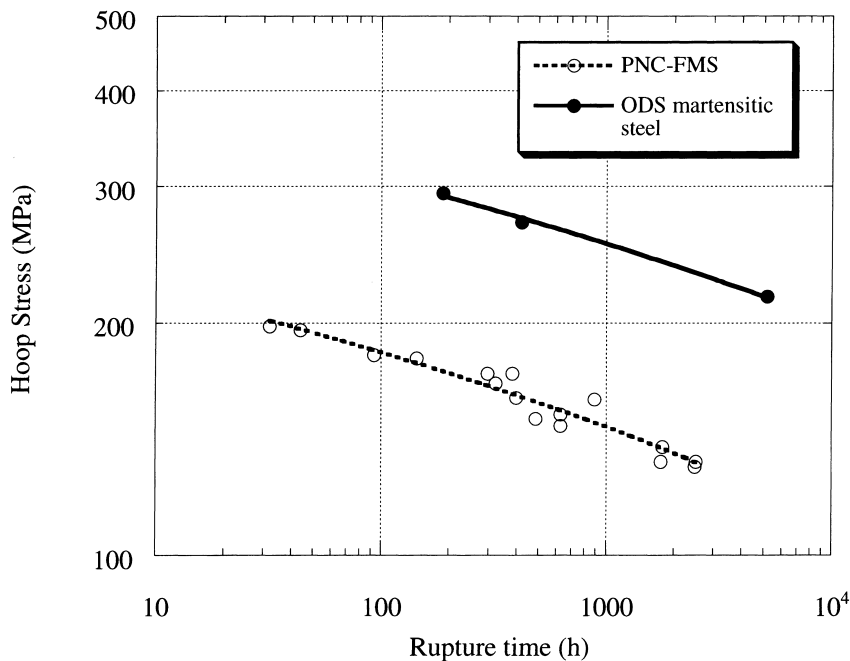


Fig. 3. Creep rupture properties of 11Cr ODS martensitic steel tube, compared with that of the usual ferritic/martensitic steel (FNC-FMS).

973 K reduces the hardness to around 380 HV. However, tempering time up to 25 h hardly affects the hardness reduction. This reason could be attributed to

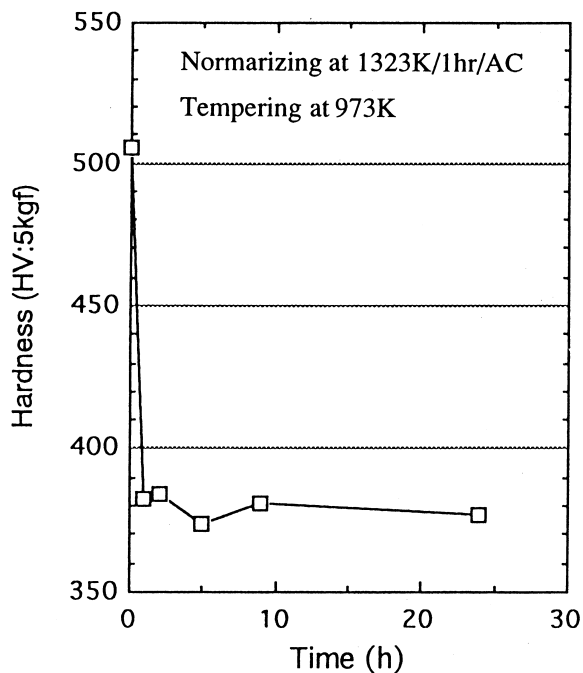


Fig. 4. Effect of normalizing and tempering condition on hardness of 11Cr ODS martensitic steel.

suppression of dislocation recovery due to pinning by the finely distributed Y_2O_3 particles. It is thus considered that the cladding manufacturing of the ODS martensitic steels by cold-rolling should not be feasible even under the normalized-tempered condition.

Therefore, the morphology and hardness changes by using a slower cooling rate of the ODS martensitic steel from the normalized condition was studied, based on the extensive investigation of the continuous cooling transformation (CCT) diagram of 11Cr-0.2C-2.8W-0.42Ti-0.36 Y_2O_3 (mass%) ODS martensitic steel. It is considered that the ferritic structure included in the ODS martensitic steel should reduce the hardness, and this technique could be important for cold-rolling manufacturing of the ODS martensitic cladding tubes.

4. Conclusion

In order to apply the ODS ferritic/martensitic steels for future FBR fuel cladding, extensive research and development has been conducted. Controlling the grain morphology by means of recrystallization processing and martensitic phase transformation successfully provided improved strength and ductility in the hoop direction of ODS ferritic/martensitic steels. The cold-rolling manufacturing of ODS cladding tubes at room temperature should be also feasible by applying the recrystallization technique.

References

- [1] T. Okuda, S. Nomura, S. Shikakura, K. Asabe, S. Tanoue, M. Fujiwara, in: A.H. Clauter, J.J. deBarbadillo (Eds.), *Solid State Powder Processing*, The Minerals, Metals and Materials Society, 1990, p. 195.
- [2] S. Nomura, T. Okuda, S. Shikakura, M. Fujiwara, K. Asabe, in Ref. [1], p. 203.
- [3] S. Ukai, M. Harada, H. Okada, M. Inoue, S. Nomura, S. Shikakura, K. Asabe, T. Nishida, M. Fujiwara, *J. Nucl. Mater.* 204 (1993) 65.
- [4] S. Ukai, M. Harada, H. Okada, M. Inoue, S. Nomura, S. Shikakura, T. Nishida, M. Fujiwara, K. Asabe, *J. Nucl. Mater.* 204 (1993) 74.
- [5] H. Okada, S. Ukai, M. Inoue, *J. Nucl. Sci. and Technol.* 33 (12) (1997) 936.
- [6] S. Ukai, T. Nishida, H. Okada, T. Okuda, M. Fujiwara, K. Asabe, *J. Nucl. Sci. and Technol.* 34 (3) (1997) 256.
- [7] S. Ukai, T. Nishida, T. Okuda, T. Yoshitake, *J. Nucl. Sci. and Technol.* 35 (4) (1998) 294.
- [8] S. Ukai, T. Nishida, H. Okada, M. Inoue, M. Fujiwara, T. Okuda, K. Asabe, *Inter. Symp. on Material Chemistry in Nuclear Environment*, 14–15 March 1996, Tsukuba, Ibraki, Japan, p. 891.
- [9] S. Shikakura, S. Nomura, S. Ukai, I. Seshimo, Y. Kano, Y. Kuwajima, T. Ito, K. Tsutaki, T. Fujita, *Nihon-Gen-shiryoku-Gakkai Shi (J. At. Energy Soc. Japan)* 33 (12) (1991) 1157 (in Japanese).