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**To cite this article:** K. Shiba, H. Tanigawa, T. Hirose & T. Nakata (2012) Development of the Toughness-Improved Reduced-Activation F82H Steel for DEMO Reactor, Fusion Science and Technology, 62:1, 145-149, DOI: [10.13182/FST12-A14127](https://doi.org/10.13182/FST12-A14127)

**To link to this article:** <https://doi.org/10.13182/FST12-A14127>



Published online: 20 Mar 2017.



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# DEVELOPMENT OF THE TOUGHNESS-IMPROVED REDUCED-ACTIVATION F82H STEEL FOR DEMO REACTOR

K. SHIBA,<sup>a</sup> H. TANIGAWA,<sup>b\*</sup> T. HIROSE,<sup>c</sup> and T. NAKATA<sup>b</sup>

<sup>a</sup>Japan Atomic Energy Agency, Tokai, Ibaraki, Japan

<sup>b</sup>Japan Atomic Energy Agency, Rokkasho, Aomori, Japan

<sup>c</sup>Japan Atomic Energy Agency, Naka, Ibaraki, Japan

Received October 14, 2011

Accepted for Publication March 29, 2012

*A toughness-improved type of F82H steel called F82H mod3 has been developed, and the material properties and irradiation behavior have been examined. The significant modification of the chemical composition is the reduction of Ti (<10 ppm) and N (<20 ppm) as impurities and the increase of Ta (0.1%) as an alloying element. The ductile-to-brittle transition temperature (DBTT) is improved to  $-90^{\circ}\text{C}$  from  $-45^{\circ}\text{C}$  for F82H IEA without change in strength. However, the creep rupture time of F82H mod3 was 1/10 of F82H IEA. Another feature of the F82H mod3 is the stability of the material properties. Higher temperature normalization ( $1080^{\circ}\text{C}$ ) degrades the*

*DBTT only to  $-80^{\circ}\text{C}$  due to grain coarsening without large change in strength. It is quite important for large-scale production of the material in high quality. Preliminary neutron irradiation experiments up to 17 dpa showed better irradiation resistance to changes in fracture toughness than F82H IEA.*

**KEYWORDS:** steels ferritic/martensitic, mechanical properties, structural materials

*Note: The figures in this paper are in color only in the electronic version.*

## I. INTRODUCTION

Reduced-activation ferritic/martensitic (RAF/M) steel is one of the candidate structural materials for blanket of a DEMO fusion reactor. RAF/M steels are designed to reduce the radiation-induced radioactivity. In the long-term R&D program of the blanket established by the Fusion Council of Japan in 1999, a ceramic breeder blanket with reduced-activation ferritic/martensitic (RAF/M) steel is regarded as the near-term candidate concept of the blanket for a fusion power demonstration plant in Japan.<sup>1</sup> One of the most important issues on RAF/M steels is toughness loss and degradation of ductile-to-brittle transition temperature (DBTT) due to irradiation hardening below  $400^{\circ}\text{C}$ .<sup>2–4</sup> Because this toughness loss strongly affects the blanket life-time, a toughness-improved type of F82H steel called F82H mod3 has been developed and the material properties and irradiation behavior has been examined. These

results are reflected in the material specification for Japanese DEMO reactor development.

## II. EFFECT OF ALLOYING ELEMENTS ON TOUGHNESS

Six alloys with different amounts of Ta and Ti were prepared to investigate the effects of these elements on toughness. Chemical compositions of these alloys (F1–F6) are listed in Table I with previous three F82H large heat (5 tons) products, F82H 1st heat (pre-IEA heat) and F82H 2nd/3rd heats (IEA heats). F1–F6 alloys were normalized at  $1040^{\circ}\text{C}$  for 30 min (air-cooled) and then tempered at  $740^{\circ}\text{C}$  and  $780^{\circ}\text{C}$  for 90 min followed by air-cooling (AC). Thermal treatment conditions of F82H 1st, 2nd, and 3rd alloys are  $1040^{\circ}\text{C} \times 40$  min AC +  $740^{\circ}\text{C} \times 120$  min AC,  $1040^{\circ}\text{C} \times 40$  min AC +  $750^{\circ}\text{C} \times 60$  min AC and  $1040^{\circ}\text{C} \times 40$  min AC +  $750^{\circ}\text{C} \times 60$  min AC, respectively.<sup>5,6</sup> Grain-sizes of these alloys are summarized in Figure 1 as a function of Ta content. Grain-size is shown as ASTM grain-size number in this figure.<sup>7</sup>

\*E-mail: tanigawa.hiroyasu@jaea.go.jp

TABLE I  
Chemical Composition of Experimental Alloys\*

	Fe	Cr	W	V	Ta	C	Ti	Si	Mn	P	S	Al	N
F1	Bal.	7.83	1.91	0.19	0.04	0.095	0.008	0.11	0.49	0.0012	0.0032	0.017	0.0082
F2	Bal.	7.86	1.92	0.19	0.04	0.095	0.010	0.11	0.49	0.0010	0.0032	0.020	0.0084
F3	Bal.	7.88	1.91	0.19	0.04	0.096	0.030	0.11	0.48	0.0011	0.0033	0.019	0.0083
F4	Bal.	7.08	1.96	0.20	0.12	0.094	0.005	0.11	0.49	0.0010	0.0033	0.020	0.0092
F5	Bal.	8.02	1.95	0.20	0.12	0.095	0.010	0.12	0.50	0.0008	0.0032	0.018	0.0095
F6	Bal.	8.01	1.96	0.20	0.12	0.096	0.030	0.12	0.49	0.0007	0.0033	0.019	0.0097
F82H 1st(pre-IEA)	Bal.	7.46	2.10	0.18	0.03	0.097	0.008	0.09	0.07	0.002	0.003	0.014	0.004
F82H 2nd (IEA heat)	Bal.	7.71	1.95	0.16	0.02	0.090	0.010	0.11	0.16	0.002	0.002	—	0.006
F82H 3rd (IEA heat)	Bal.	7.87	1.98	0.19	0.03	0.090	0.004	0.07	0.10	0.003	0.001	—	0.007

\*In mass percentage.

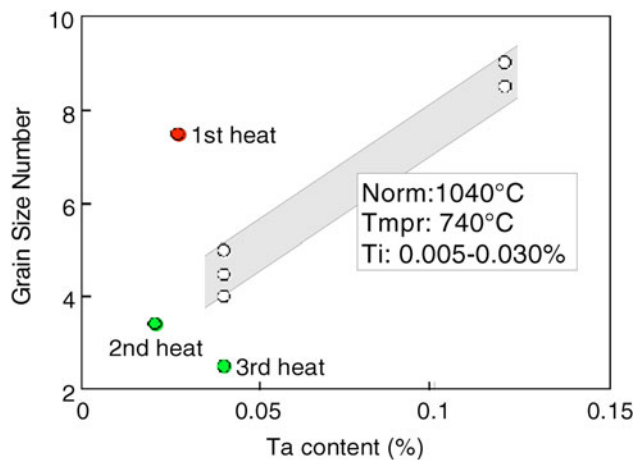


Fig. 1. Grain size of alloys as a function of Ta content.

This figure indicates Ta content strongly affects the grain-size. Figure 2 shows the DBTT obtained by Charpy impact test using full-size ( $10 \times 10 \times 55 \text{ mm}^3$ ) or half-size ( $10 \times 5 \times 55 \text{ mm}^3$ ) V-notched specimens. As shown in this figure, smaller grain-size gives better DBTT. DBTT of alloys are presented in Figure 3 as a function of Ti content. This figure shows DBTT is dramatically improved by the reduction of Ti to less than 0.01%. Microstructural observation indicates the sensitivity to Ti content is mainly caused by the formation of complex Ti nitride and carbide,  $\text{Ti}(\text{N}, \text{C})$  precipitates. This means reduction of Ti and N is important to improve the toughness of the material. F82H 1st heat contains 0.010% Ti, but DBTT of this material was low. This material contains only 0.0035% N, so that TiN precipitation might be suppressed. These results can be interpreted to suggest  $\text{Ti} < 0.010\%$ , and  $\text{N} < 0.004\%$  to improve the toughness. Reduction of N is also effective to reduce the irradiation-induced radioactivity. Additionally, Ta should be increased to  $\sim 0.1\%$  to obtain

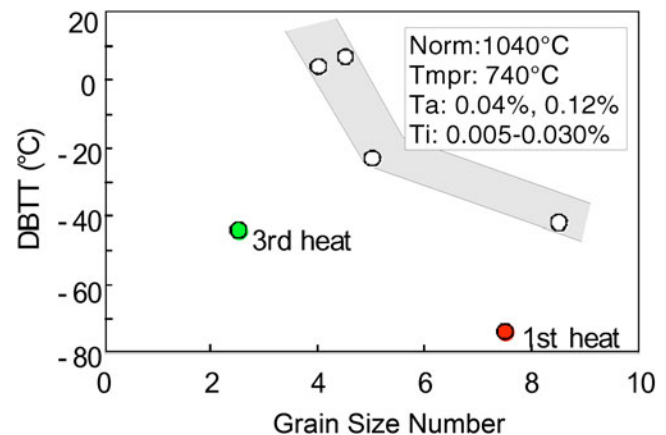


Fig. 2. Ductile-to-brittle transition temperature (DBTT) of alloys as a function of grain-size.

smaller grain-size. This alloy specification is defined as F82H mod3.

### III. F82H MOD3

Two batches (batch 1 and batch 2) of F82H mod3 were fabricated and tested. Chemical compositions of these alloys are listed in Table II. The stability of the microstructure and DBTT on F82H mod3 (batch 1) has been reported previously.<sup>5</sup> F82H mod3 maintains small grain-size and low DBTT for a range of normalizing temperature between 1000 and 1060°C. F82H mod3 is also quite stable against temperature variation during the hot-rolling process. F82H mod3 batch 2 was normalized at two different conditions, 1040°C and 1080°C. The grain-size and Vickers' hardness (98 N of test-load) of batch 1 and batch 2 were quite similar for 1040°C normalized materials. F82H mod3 batch 2 1080°C normalized material showed ten times larger grain-size than that of

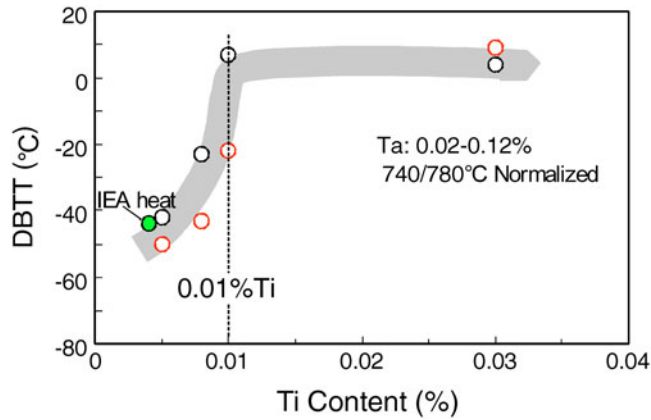


Fig. 3. Ductile-to-brittle transition temperature (DBTT) of alloys as a function of Ti content.

1040°C normalized, but it is still the same size as F82H IEA. It means TaC dissolve at this temperature. TaC solubility is given by Tamura et al.<sup>8</sup> Their result gives solubility limit of 1070°C for 0.1%Ta (F82H mod3) and 990°C for 0.04%Ta (F82H IEA). The results mean grain-size is controlled by TaC precipitation, and 1040°C of normalizing temperature is too high for F82H IEA considering the toughness.

Tensile and Charpy impact properties are shown in Figure 4 comparing with F82H IEA. Tensile tests were performed using round-bar tensile specimens ( $\phi 6 \times 36 \text{ mm}^3$  in gage section) at room temperature and Charpy impact tests were carried out using full-size ( $10 \times 10 \times 55 \text{ mm}^3$ ) or half-size ( $10 \times 5 \times 55 \text{ mm}^3$ ) V-notched specimens. F82H mod3B means F82H mod3 batch 2 normalized at 1080°C. In spite of the difference in

TABLE II

Chemical Composition of F82H mod3 and F82H mod3+N\*

	Fe	Cr	W	V	Ta	C	N	Ti
F82H mod3(batch1)	Bal.	8.16	1.94	0.20	0.092	0.100	0.0014	<0.001
F82H mod3(batch2)	Bal.	7.94	1.89	0.21	0.10	0.101	0.0015	<0.001
F82H mod3+N	Bal.	7.92	1.87	0.21	0.10	0.098	0.0085	<0.001

\*In mass percentage.

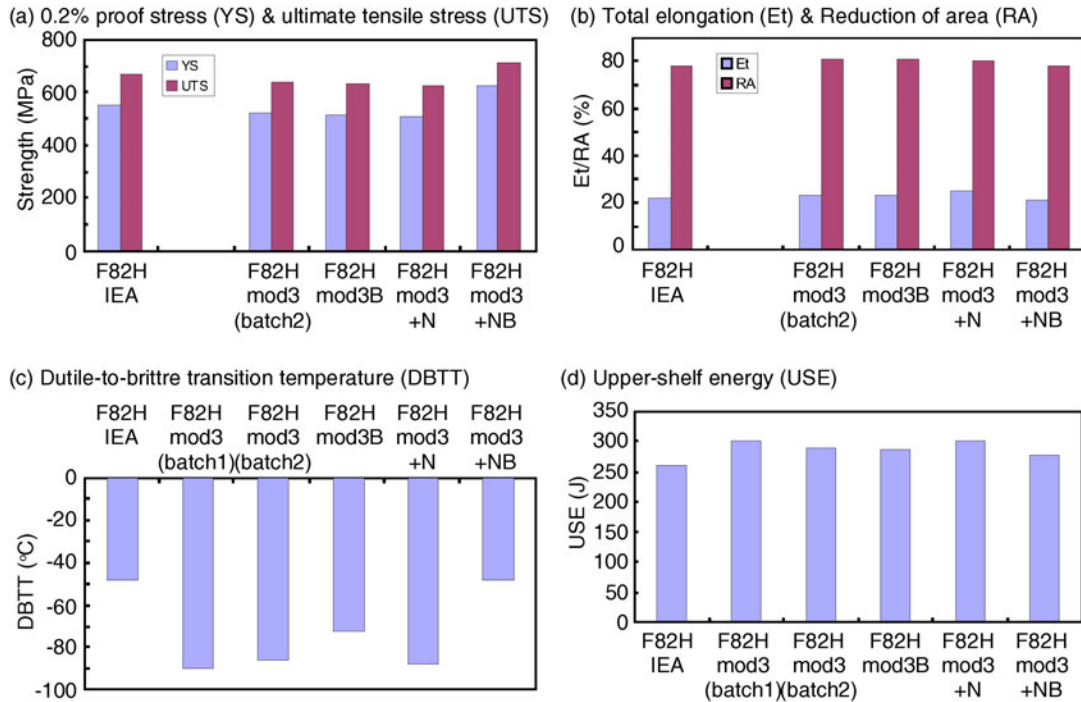


Fig. 4. Tensile and Charpy impact properties of F82H mod3 and F82H mod3+N.

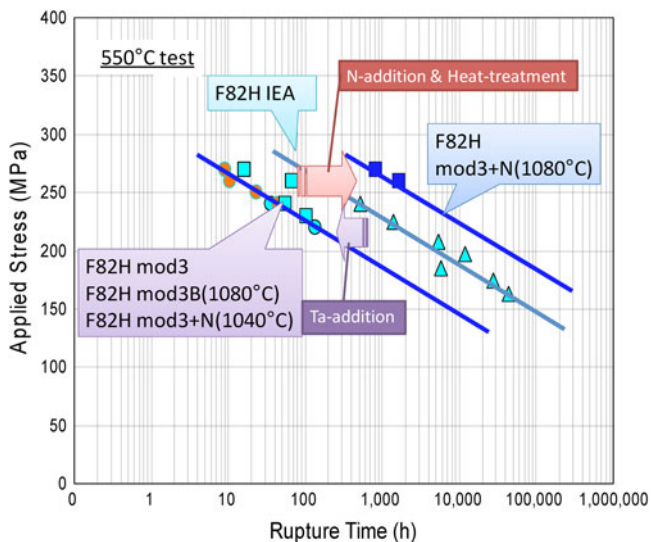


Fig. 5. Creep rupture properties of F82H mod3 and F82H mod3+N.

grain-size, F82H mod3 has the same tensile and Charpy properties in both 1040°C and 1080°C normalized conditions. F82H mod3 has slightly lower strength and larger elongation than F82H IEA, but they have almost the same tensile property. F82H mod3 has quite low DBTT ( $-85 \sim -90^{\circ}\text{C}$ ) compared to F82H IEA ( $-45 \sim -50^{\circ}\text{C}$ ) and about 15% larger Upper-shelf energy (USE) than F82H IEA. The difference of grain-size due to normalizing temperature caused about  $20^{\circ}\text{C}$  of increase in DBTT and 3% of decrease in USE, but they are still remarkably better than F82H.

Creep rupture tests of F82H mod3 were also performed using round-bar type specimens ( $\phi 4 \times 20 \text{ mm}^3$  in gage section) at  $550^{\circ}\text{C}$  in air. The test results are shown in Figure 5. As shown in this figure, gradient of the creep curve was similar in both F82H mod3 and F82H IEA, but creep rupture time of F82H mod3 was about 1/10 of F82H IEA. Usually smaller grain-size causes lower creep

strength, but larger grain-size due to  $1080^{\circ}\text{C}$  normalizing (F82H mod3B) showed the same creep strength of smaller grain-size (F82H mod3). It seems grain-size does not affect the creep strength at  $550^{\circ}\text{C}$  on this type of material. These results suggest Ti(C, N) has a large effect on maintaining the creep strength on F82H IEA.

#### IV. EFFECT OF N ON F82H MOD3

Because F82H mod3 has lower creep strength than F82H IEA, N addition was examined. Chemical composition, heat treatment condition, grain-size, and Vickers hardness of N-added F82H mod3 (F82H mod3+N) are listed in Tables II and III. 85 ppm of N was added to F82H mod3, but it is still a high purity level considering a large scale production, which usually contains 100~200 ppm of N. Two different normalizing temperatures, 1040 and  $1080^{\circ}\text{C}$  were applied to this material also.

Tensile, Charpy impact test and Creep rupture test results of F82H mod3+N are shown in Figure 4 and Figure 5. Specimen size and test condition were the same as F82H mod3. F82H mod3+N normalized at  $1040^{\circ}\text{C}$  is indicated as F82H mod3+N, and F82H mod3+N normalized at  $1080^{\circ}\text{C}$  is indicated as F82H mod3+NB. These results indicate that F82H mod3+N has completely the same properties as F82H mod3 when it is normalized at  $1040^{\circ}\text{C}$ . However, F82H mod3+N normalized at  $1080^{\circ}\text{C}$  showed higher strength and less toughness than F82H mod3. Yield and ultimate tensile strength was 15% higher and DBTT was the same level as F82H IEA. The most remarkable difference appeared in creep strength. Creep rupture time increased about 100 times longer than F82H mod3, and it is 10 times longer than F82H IEA. High creep strength of this material is considered to be caused by TaN precipitation. Because TaC dissolves above  $1070^{\circ}\text{C}$ , free Ta can be form TaN. These results mean that F82H mod3+N has two different characters, high toughness and high creep strength with different heat treatment conditions. Furthermore, control of the impurity level

TABLE III  
Heat Treatment Conditions, Grain Size, and Vickers Hardness (Hv) of Alloys

	Normalizing	Tempering	Grain Size	Hv
F82H IEA	$1040^{\circ}\text{C} \times 38\text{min}$	$750^{\circ}\text{C} \times 60\text{min}$	$150 \mu\text{m}$	218
F82H mod3(batch1)	$1040^{\circ}\text{C} \times 30\text{min}$	$740^{\circ}\text{C} \times 60\text{min}$	$20 \mu\text{m}$	219
F82H mod3(batch2)	$1040^{\circ}\text{C} \times 30\text{min}$	$740^{\circ}\text{C} \times 60\text{min}$	$15 \mu\text{m}$	210
	$1080^{\circ}\text{C} \times 30\text{min}$	$740^{\circ}\text{C} \times 60\text{min}$	$180 \mu\text{m}$	207
F82H mod3+N	$1040^{\circ}\text{C} \times 30\text{min}$	$740^{\circ}\text{C} \times 60\text{min}$	$15 \mu\text{m}$	207
	$1080^{\circ}\text{C} \times 30\text{min}$	$740^{\circ}\text{C} \times 60\text{min}$	$150 \mu\text{m}$	233



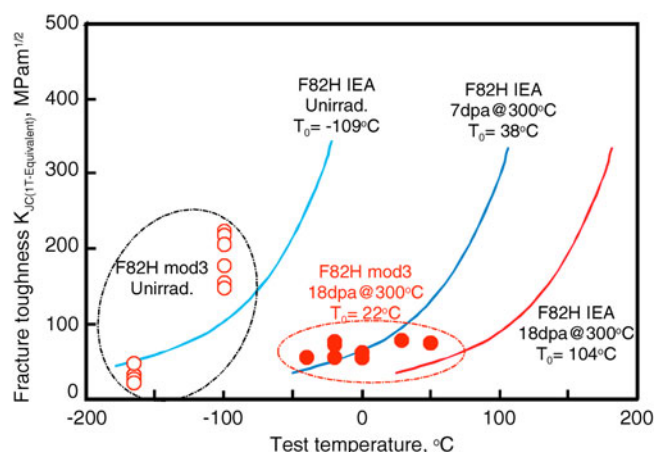


Fig. 6. Fracture toughness of F82H mod3 and F82H IEA after neutron irradiation at 300°C.

and thermal treatment temperature are quite important to maintain the quality of material.

## V. IRRADIATION RESULTS OF F82H MOD3

Preliminary neutron irradiation of F82H mod3 has been achieved up to 18 dpa using the High Flux Isotope Reactor (HFIR) at the Oak Ridge National Laboratory. Tensile tests using SSJ3 type sheet tensile specimens and fracture toughness tests using pre-cracked CVN (PCCVN) miniature size specimens were carried out after 7 and 18 dpa irradiations at 300°C. Tensile results of F82H mod3 were almost same as F82H IEA. However, degradation in fracture toughness F82H mod3 was considerably improved in F82H mod3. Figure 6 shows fracture toughness obtained by PCCVN as a function of test temperature comparing with F82H IEA results. The toughness of F82H mod3 after 18 dpa was almost same as that of F82H IEA after 7 dpa. It seems F82H mod3 has better resistance to irradiation-induced changes in toughness than F82H IEA. Irradiation to higher dose level is also scheduled.

## VI. SUMMARY

Optimization of F82H was carried out to improve its toughness and stability. Obtained results are summarized as follows:

1. High toughness type of F82H, F82H mod3 has been developed by optimization of alloying elements and impurity control.

2. F82H mod3 has better toughness than F82H IEA without loss of tensile properties.

3. The properties of F82H mod3 are quite stable and deviation of heat-treatment condition does not much affect the properties.

4. Negative effect of F82H mod3 is reduction in creep strength. Creep rupture time is  $\sim 1/10$  of F82H IEA, and grain-size does not affect creep strength on F82H mod3.

5. Creep strength of F82H mod3 can be improved by some nitrogen addition with appropriate heat-treatment condition. This improvement gives 10 times longer creep rupture time than F82H IEA.

6. Neutron irradiation to 18 dpa at 300°C has been achieved. F82H mod3 has better fracture toughness than F82H IEA in spite of the same level of irradiation hardening.

## ACKNOWLEDGMENTS

The authors wish to thank Mr. Y. Kuriki, former Kokan Keisoku co. for the fabrication of the material and testing. We also wish to thank Dr. R. E. Stoller, and other staff members in the Oak Ridge National Laboratory for our collaborative research on fusion reactor materials.

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