1.1 FABRICATION OF LOW ACTIVATION FERRITIC-MARTENSITIC STEELS CONTAINING ^{54}FE ISOTOPE—Y. Yamamoto (Oak Ridge National Laboratory)

OBJECTIVE

The goal of this task is to evaluate the effect on the irradiation microstructure and mechanical properties of the 9Cr base reduced activation ferritic-martensitic (RAFM) steels, such as EUROFER 97 and cast nanostructured alloys (CNA), prepared by using ^{54}Fe isotope. The ^{54}Fe was recovered from oxide powder that KIH provided. The ^{54}Fe isotope should accelerate the helium bubble formation during fast neutron exposure at HFIR, partially simulating the high dose irradiation conditions within relatively short exposure times.

SUMMARY

Four trial EUROFER97 steel plates were successfully fabricated by arc-melting with natural Fe and hot-rolling with a steel guide. The arc-melting was conducted to measure the yield of each alloying element during the melt process, and the trial hot-rolling with a steel guide was performed to produce steel plates without barreling. Based on the process parameter of the trial heats, two RAFM steel plates (targeted to be EUROFER97 and CNA) made of ^{54}Fe isotope powder, with size ~0.25 x 1.4 x 2.5 inches, were successfully fabricated. The detailed planning of the machining of SS-J3 tensile specimens and M4 bend bar specimens, as well as developing an irradiation test plan for HFIR, is currently in progress.

PROGRESS AND STATUS

Introduction

There are no test facilities available with a neutron spectrum that matches D-T fusion. Various methods are used to simulate fusion reactor conditions and evaluate irradiation effects in structural materials for fusion reactors. For reduced-activation ferritic-martensitic (RAFM) steels, alloys doped with B or Ni have been used in fission test reactors to produce helium through (n, α) reactions while the fast neutrons produce displacement damage. However, both B and Ni tend to segregate at grain boundaries, compromising the simulation of uniform irradiation damage. It has been proposed to replace all natural Fe with the isotope ^{54}Fe in selected RAFM steels to expect acceleration of the He transmutation and resulting bubble formation dispersed uniformly within relatively short period of irradiation time. This was previously attempted in the High Flux Isotope Reactor (HFIR) with ^{55}Fe by Liu et al. [1]. HFIR neutron irradiation will achieve simultaneous He and dpa production throughout the bulk of specimens in a damage evaluation study.

About 400 g of mixed oxide steel powder was delivered from Karlsruhe Institute of Technology (KIT) which consisted primarily of ^{54}Fe together with small amounts of Cr, W, and other alloying elements. The origin of the powder material was a trial heat of EUROFER 97 with ^{54}Fe made at KIT. However, the heat was accidentally contaminated with high amount of B and C so that the material was fully dissolved in an acid and turned into oxide powders during the recovery process [2]. Reduction of the oxide powders was used at ORNL, in order to extract the ^{54}Fe with reasonably low oxygen content. Total ~220 g of ^{54}Fe powder containing small amounts (~1wt.%) of Cr and W and 79 wppm of oxygen was obtained in this effort in CY2015 [3].

The powder was used for fabricating two different RAFM steels; EUROFER97 and CNA steels, and two plates of the alloys were prepared. In this report, the on-going fabrication effort of the RAFM steels with ^{54}Fe, together with trial RAFM steels with natural Fe, is summarized.

Experimental Procedures

A total of four trial reference RAFM steel ingots (with compositions targeted equal to EUROFER97 steel, ~110g for each) were arc-melted with natural Fe and pure element feedstock. Commercial CrN/Cr_2N powder was used for the nitrogen source of the ingots. A new Cu mold with size 0.5 x 1.4 x 1.4 inches was machined to produce square cast ingots, as shown in Figure 1. The ingots were homogenized at 1150°C, and then hot-rolled at 1040°C, with a guide steel plate in order to fabricate plates with target geometry ~0.25 x 1.4 x 2.5 inches. The plate samples were normalized at 1040°C for 10 min and then air-cooled to room temperature. The samples were tempered at 760°C for 1.5 h, and then air-cooled to room temperature.

The chemical compositions of the trial ingots were measured by ICP-OE/IGF analyses. The results of the first two trial ingots were used to calculate the yield of each element, and the second two trial ingots were melted with the calibrated alloy compositions. After confirming the calibration results of the second cast ingots, two RAFM steels (E97-54 and CNA-54, equivalent to EURFER97 and CNA steels, respectively) were arc-melted with the ^{54}Fe powder. The cast ingots were hot-rolled and heat-treated in the same way of the trial ingots.

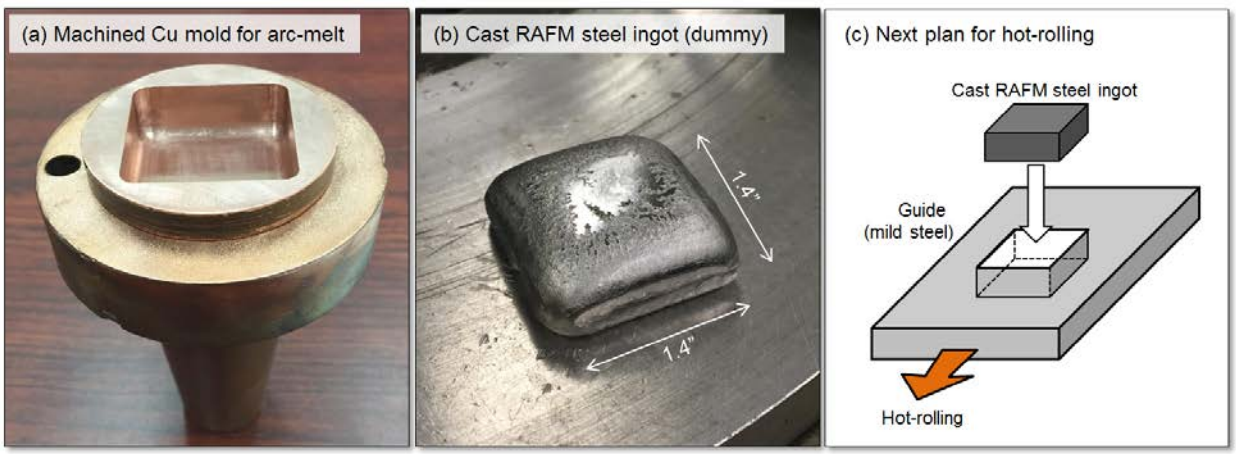

Figure 1. (a) Newly machined Cu mold, (b) a cast trial RAFM steel ingot, (c) and an illustration of the next plan (hot-rolling) to be conducted.

Results

Table 1 summarizes the analyzed chemical compositions of the trial EUROFER97 heats. The results indicated that two trial heats, E97-03 and E97-05, were out of the specification, and it was found that the W content of the heats were mistakenly selected lower than the target nominal content (1.1 wt.%). It was also found that the other elements, especially Mn, C, and N were much lower than those expected from the targets. The loss of the N content was due to the nitrogen gas bubbling during the arc-melting process, which was visually observed during the arc-melt operation. The number of arc-melting cycles (to ensure homogeneous mixing of the ingot) was also important to preserve the nitrogen content in the heat. The yields of these elements were calculated, and the calibrated amounts of the alloying additions were used for two newly arc-melted trial heats (#22 and #23). By minimizing the number of arc-melts, the resultant alloy compositions mostly met the target specification. Only the C content was higher than the target, so that re-calibration of the carbon was used for the next melt with the ^{54}Fe powder.

Table 1. Analyzed compositions of trial EUROFER97 heats melted at ORNL

Name	Composition, wt.%								Remarks
	Fe	C	Mn	Cr	V	Ta	W	N	
Target (for E97-03/05)	89.20	0.11	0.40	9.00	0.20	0.12	0.91	0.060	The target W content was incorrect
E97-03	Bal.	0.077	0.36	8.84	0.19	0.11	0.84	0.008	Arc-melted x10
E97-05	Bal.	0.077	0.37	8.83	0.18	0.11	0.87	0.014	Arc-melted x7
Target (for new heats)	88.94	0.11	0.50	9.00	0.20	0.12	1.10	0.030	Nominal (revised)
Heat #22	Bal.	0.14	0.51	8.95	0.21	0.13	1.1	0.021	Arc-melted x4
Heat #23	Bal.	0.13	0.51	8.94	0.21	0.13	1.11	0.022	Arc-melted x4

The arc-melted trial ingot with size 0.5 x 1.4 x 1.4 inches was successfully rolled into plate with size of 0.24 x 1.42 x 2.50 inches, without the formation of any edge cracks or side barreling, as shown in Figure 2. It was homogenized, hot-rolled at 1040°C, and then normalized at 1040°C for 30 min and tempered at 760°C for 90 min, with the guide steel plate. An optical micrograph of the trial EUROFER97 heat, Figure 2c, showed a uniform, full tempered martensitic microstructure without any anisotropic features (e.g. elongated grains along the rolling direction), suggesting that material fabrication process was successful.

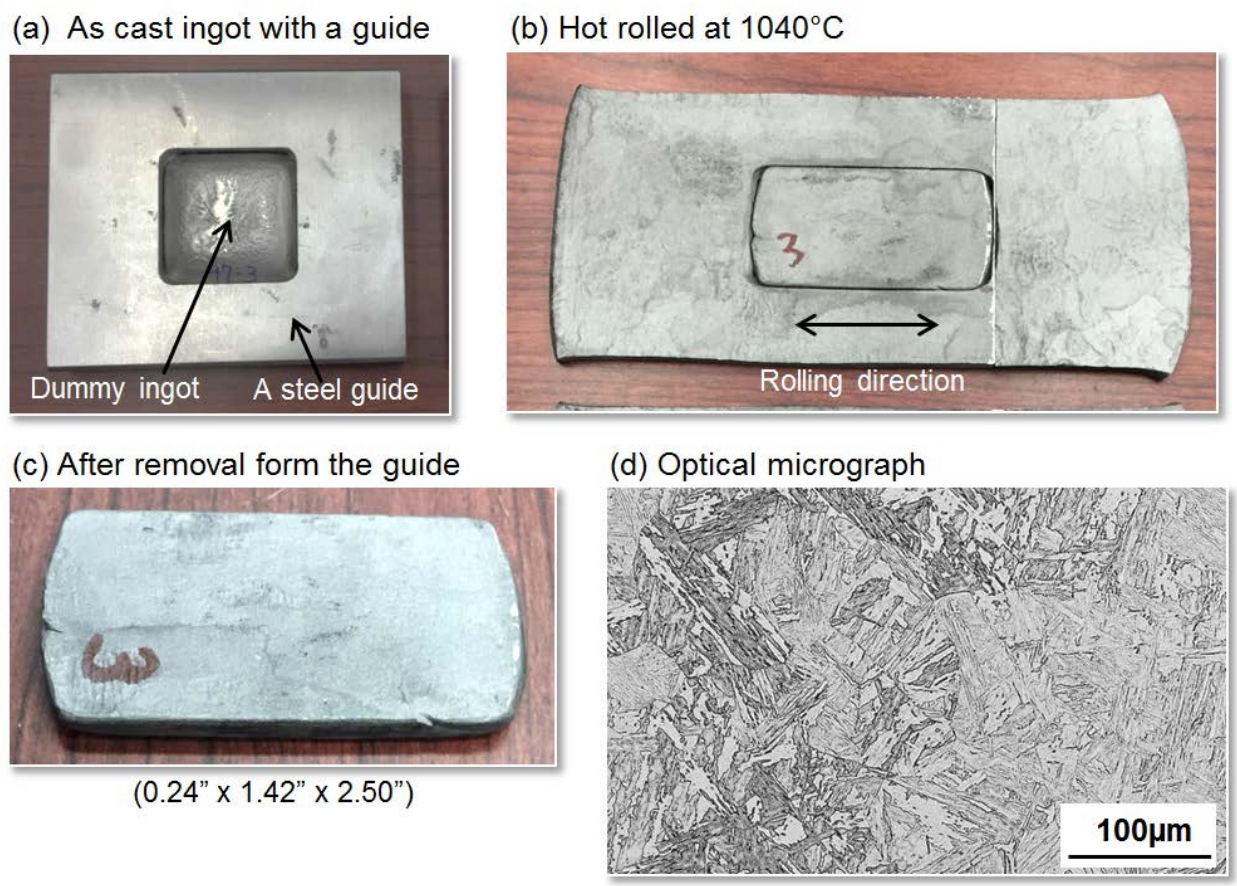

Figure 2. (a) As-cast trial ingot with a mild steel guide, (b) the rolled plate with the guide, (c) the rolled plate after removal from the guide, and (d) the microstructure after the heat treatment.

Two new heats of EUROFER97 and CNA steels were arc-melted with ^{54}Fe isotope powder. The arc-melted ingots (0.5 x 1.4 x 1.4 inches, Figure 3a) were successfully rolled to plate with size 0.24 x 1.42 x 2.50 inches, without any edge cracks or side barreling (Figure 3b). They were homogenized, hot-rolled, and then normalized and tempered. There were no edge cracks or side barreling, as with the trial EUROFER97 heat fabrication.

Discussion of the machining plan to prepare SS-J3 tensile specimens and M4 bend bar specimens is in progress. The current plan is to machine a total of 18 SS-J3 and 16 M4 bend bar specimens from each plate, as shown in Figure 4. The detailed irradiation test plans for HFIR irradiation experiments as well as the required specimen numbers will soon be finalized.

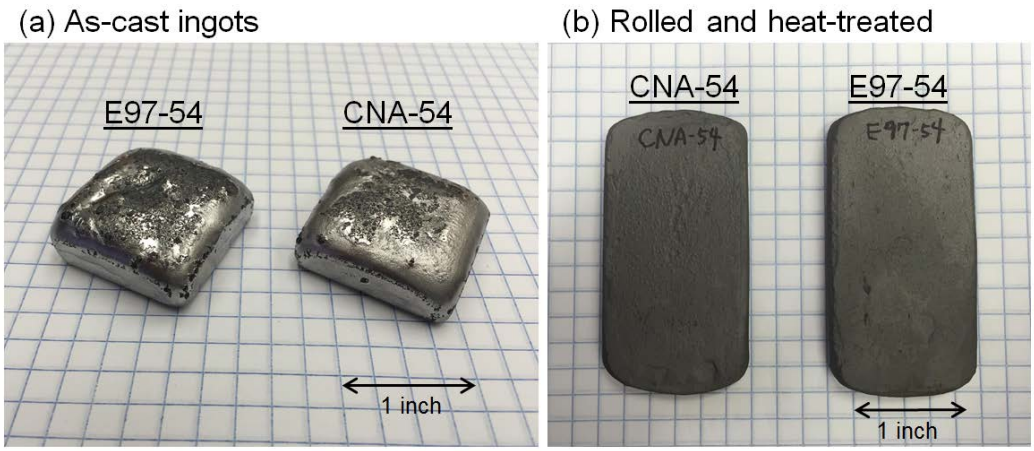

Figure 3. (a) As-cast ingots of EUROFER97 (E97-54) and CNA (CAN-54) made with ^{54}Fe isotope, and (b) the rolled and annealed plates.

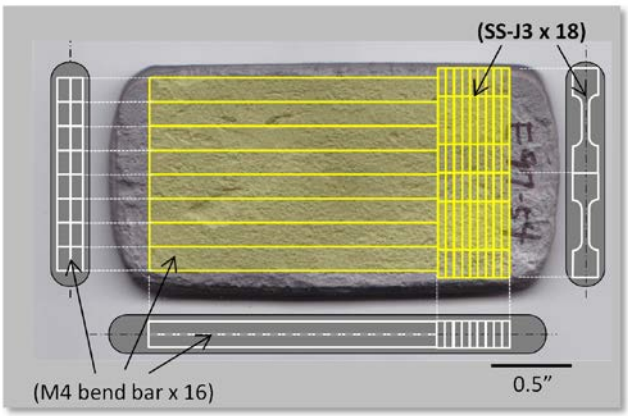

Figure 4. Machining plan for SS-J3 and M4 bend bar specimens.

References

[1] H. Liu, M. A. Abdou, L. R. Greenwood, ^{55}Fe effect on enhancing ferritic steel He/dpa ratio in fission reactor irradiations to simulate fusion conditions, Fusion Engineering and Design, 88 (2013) 2860-2864.

[2] M. Rieth et al., EUROFER-Fe54 Specimen Production and Irradiation Proposal, TW4-TTMS-001 D01,TW5-TTMS-001 D02, Karlsruhe Institute of Technology Interner Bericht, Projekt Fusion 31.40.03, Dec. 2010

[3] Y. Yamamoto et al., Fabrication of Low Activation Ferritic-Martensitic Steels Containing ^{54}Fe Isotope, poster presentation at ICFRM-17, Aachen, Germany, October 2015.