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Author(s): Maloy, Stuart Andrew
Pestovich, Kimberly Shay
Anderoglu, Osman
Aydogan, Eda

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Development of New Heats of Advanced Ferritic/Martensitic Alloys

Fuel Cycle Research & Development

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Stuart A. Maloy, Osman Anderoglu, Eda Aydogan, Kim Pestovich
Los Alamos National Laboratory

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SUMMARY

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The Fuel Cycle Research and Development program is investigating methods of transmuting minor actinides in various fuel cycle options. To achieve this goal, new fuels and cladding materials must be developed and tested to high burnup levels (e.g. >20%) requiring cladding to withstand very high doses (greater than 200 dpa) while in contact with the coolant and the fuel.

To develop and qualify materials to a total fluence greater than 200 dpa requires development of advanced alloys and irradiations in fast reactors to test these alloys. Recent results from testing numerous ferritic/martensitic steels at low temperatures suggest that improvements in low temperature radiation tolerance can be achieved through carefully controlling the nitrogen content in these alloys. Thus, four new heats of HT-9 were produced with controlled nitrogen content: two by Metalwerks and two by Sophisticated Alloys. Initial results on these new alloys are presented including microstructural analysis and hardness testing. Future testing will include irradiation testing with ions and in reactor.

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1. Introduction

The Fuel Cycle Research and Development program is investigating methods of transmuting minor actinides in various fuel cycle options. To achieve this goal, new fuels and cladding materials must be developed and tested to high burnup levels (e.g. >20%) requiring cladding to withstand very high doses (greater than 200 dpa) while in contact with the coolant and the fuel.

To develop and qualify materials to a total fluence greater than 200 dpa requires development of advanced alloys and irradiations in fast reactors to test these alloys. Recent results from testing numerous ferritic/martensitic steels at low temperatures suggest that improvements in low temperature radiation tolerance can be achieved through carefully controlling the nitrogen content in these alloys[1]. Thus, new heats of HT-9 were produced by Metalwerks and Sophisticated Alloys with controlled nitrogen content. Initial results on these new alloys are presented including optical microscopy and hardness testing. Future testing will include irradiation testing with ions and in reactor.

2. Experimental

Four new heats of HT-9 were produced: two by Metalwerks with controlled nitrogen content between 0.004 and 0.062 wt.% nitrogen and two by Sophisticated alloys (SA-3 and SA-4) with controlled nitrogen between 0.001 and 0.044 wt. %. During the processing of the Metalwerks alloys, carbon was lost reducing it to levels below 0.20 %. Thus, to further understand the effect of the reduced carbon on the microstructure, Electron Backscatter Detection (EBSD) analysis and hardness testing was performed.

Table 1: Elemental compositions (wt%) of the two alloys with low and high impurity contents.

Element	C	Mn	Si	Ni	Cr	Mo	W	V	N	P	S	Fe
Low imp (MW-1)	.07	.62	.28	.52	11.2	1.0	.5	.3	.004	<.005	.004	Bal.
High imp (MW-2)	.14	.58	.28	.52	11.3	1.0	.5	.3	.062	<.005	.002	Bal.
SA-3	.20	.55	.25	.51	11.1	1.0	.47	.3	.001	<0.010	.001	Bal.
SA-4	.20	.56	.26	.52	11.4	1.0	.48	.3	.044	<0.010	.001	Bal.

3. Results

The newly developed HT-9 alloys with controlled interstitial content were first normalized at 1040°C for 30 minutes and then quenched in air (quartz encapsulated), tempered at 760°C for 1 and 2 hours to investigate the effect of tempering time and C and impurity content on microstructure and mechanical properties. Table 1 shows the composition of the high and low impurity alloys. Figure 1 shows the electron backscatter diffraction (EBSD) gray scale micrographs of the as-quenched and quenched+1 h annealed samples for both low and high impurity alloys. Therefore, martensite formation by quenching is possible. In the as-quenched form in Fig. 1a, there is delta ferrite at the pre-austenite grain boundaries together with retained austenite. After tempering at 760°C for 1 h, amount of the white equiaxed grains decreased indicating that unstable retained austenite transformed into a more stable phase, shown in Fig. 1b. Even though high impurity alloys have low carbon amount, alloying content leads to the shift of transformation curves to the longer times. On the other hand, low alloying content results in rapid transformations. As shown in Fig. 1c, even though the low alloy sample was quenched, a fraction of austenite was transformed to pro-eutectoid ferrite, remaining being martensite. After tempering at 760°C for 1 h, the martensite phase transformed into tempered martensite and the microstructure became coarser, as in Fig. 1d.

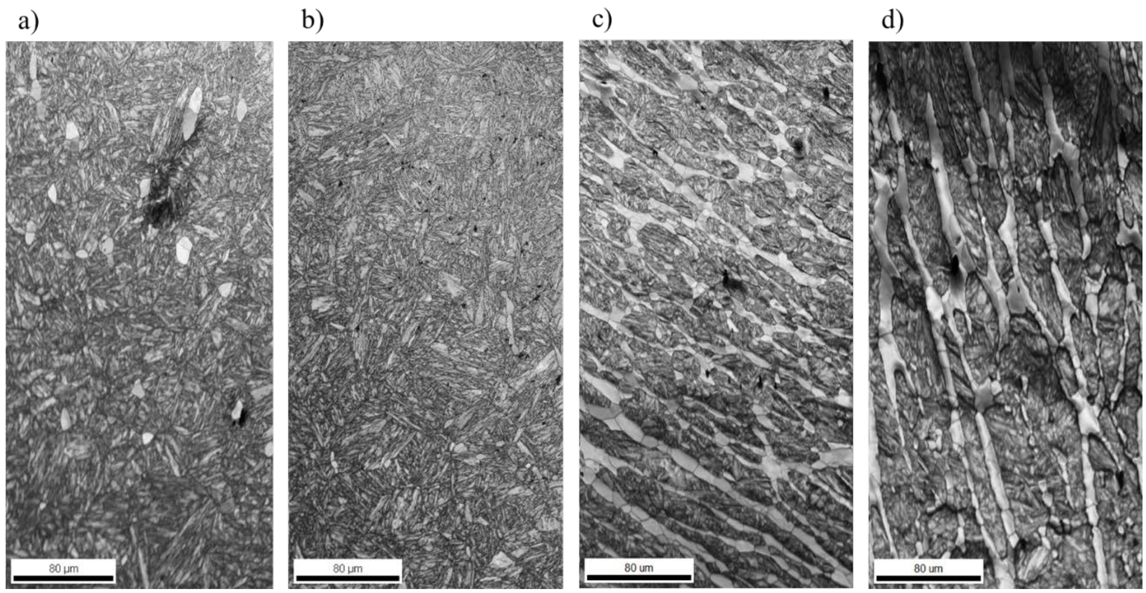

Fig. 1: EBSD gray scale maps of HT-9 samples having high amount of impurity (MW-2) and (a) air quenched, (b) air quenched and tempered at 760°C for 1 h; HT-9 samples having low amount of impurity (MW-1) and (c) air quenched, (d) air quenched and tempered at 760°C for 1 h.

Hardness evolution as a function of tempering time is shown in Fig. 2. High impurity alloys have ~100 HV higher hardness than the low impurity alloys because of the above stated microstructural differences. Tempering quickly drops the hardness initially due to tempering of martensite and formation of $M_{23}C_6$ carbides. Additional tempering doesn't seem to change the hardness significantly.

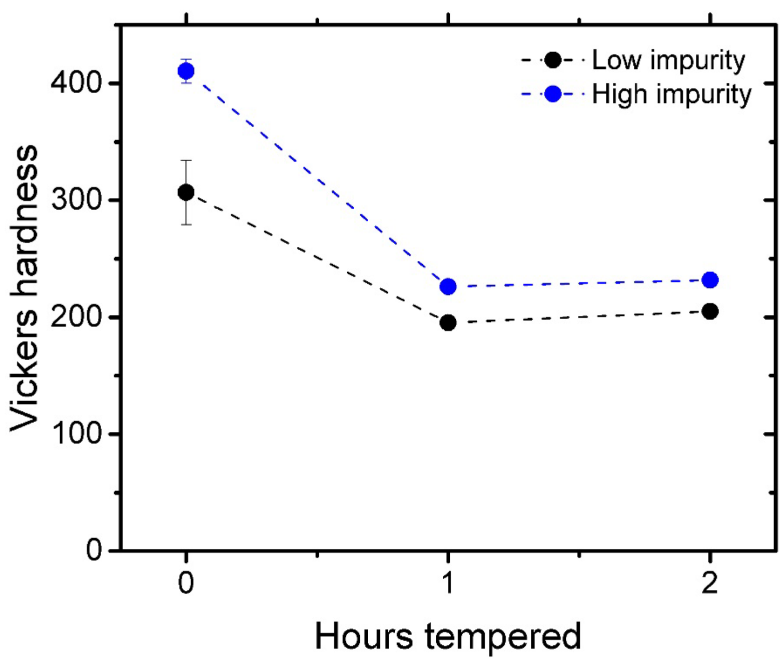

Fig. 2: Hardness vs. tempering time plot of high and low alloy Metalwerks HT-9 samples.

4. Conclusions

Four new heats of HT-9 were produced: Two by Metalwerks and two by Sophisticated Alloys with controlled nitrogen content between 0.001 and 0.064 wt. %. For the two Metalwerks HT-9 heats, some carbon was lost during processing leading to reduced carbon below the desired 0.20 wt. %. Microstructural analysis and hardness testing was performed to a reduction in hardness with reduced carbon which coincided with reduced martensite content in the reduced carbon alloys also. These four alloys will be used for future mechanical and irradiation testing on these new heats and will be compared to previous results on irradiation effects in ferritic/martensitic steels.

[1] S.A. Maloy, T.A. Saleh, O. Anderoglu, T.J. Romero, G.R. Odette, T. Yamamoto, S. Li, J.I. Cole, R. Fielding, Journal of Nuclear Materials, 468 (2016) 232-239.