## 1.1 DEVELOPMENT OF ADVANCED REDUCED ACTIVATION FERRITIC/MARTENSITIC STEELS —

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### OBJECTIVE

Develop alloy composition and thermomechanical treatment (TMT) to produce high density of nanoprecipitates, high temperature-capable, more manufacturable, and affordable reduced-activation ferritic/martensitic (RAFM) steels.

### SUMMARY

A new RAFM steel has been designed to favor the formation of MC (M = metal) carbides because a recent study has shown superior stability of TaC over TaN and VN nanoprecipitates under thermal aging, creep testing, and $Fe^{2+}$ ion irradiation at 500°C. A lab-scale heat of the new alloy has been fabricated in conventional normalization and tempering conditions. Tensile results for test temperatures up to 800°C exhibited yield strength significantly greater than P92 steel with decent total elongation. Four different TMT conditions have been developed on Grade 92 samples. One of the conditions with warm rolling after normalization followed by 2-step tempering was identified as having the best results in terms of tensile and Charpy impact properties; these conditions will be applied to the new alloy. Additionally, preliminary microstructural characterization of the high dose $Fe^{2+}$-irradiated TaC samples has produced some new observations.

### PROGRESS AND STATUS

#### Introduction

Recent investigations on the evolution of TaC, TaN, and VN nanoprecipitates under thermal aging at 600 and 700°C, creep testing at 600°C, and $Fe^{2+}$ ion irradiation up to ~49 dpa at 500°C indicated the superior stability of TaC over TaN and VN [1,2]. This finding sheds a light on developing new RAFM steels favoring the formation of MC carbides rather than nitrides. The MC-type precipitates are believed to be beneficial for maintaining high temperature strength of this class of steels. Therefore, a new alloy, designated as TT1, has been designed and prepared to test its performance. In the meantime, TMT processes have been explored to identify an appropriate process for property improvements of the alloy. Additionally, microstructural characterization of the high dose $Fe^{2+}$-irradiated samples of TaC, TaN, and VN has been initiated.

#### Experimental Procedure

Computational thermodynamics was employed to design new RAFM steels with an increased amount of MC-type precipitates. One of the new alloys, TT1, has been fabricated using vacuum arc melting and drop casting, yielding a small experimental heat (~1 lb.). Part of the cast alloy was subjected to hot rolling followed by standard normalization (N) and tempering (T) heat treatment. Several routes of TMTs involved with warm rolling (WR) have been conducted on a Grade 92 heat to investigate the effect of WR on tensile properties and Charpy impact resistance.

Tensile testing was conducted using miniature specimens (type SS-3: 25.4 x 4.95 x 0.76 mm), machined along the rolling direction of the plate, at a nominal strain rate of 0.001 $s^{-1}$. Half-size Charpy specimens were used to assess ductile-brittle transition temperature and upper shelf energy of the processed samples. The microstructures of the samples were characterized using optical microscopy, scanning electron microscopy (SEM), and transmission electron microscopy (TEM). A FEI CM200 field-emission-gun TEM/STEM equipped with an EDAX energy dispersive X-ray spectroscopy (EDS) detector was primarily used to characterize the $Fe^{2+}$-irradiated samples. TEM specimens, perpendicular to the surfaces, were lifted out and thinned to electron-transparent using focused ion beam (FIB) on a Hitachi

NB5000. Specimen thickness of the characterized regions was estimated using convergent beam electron diffraction (CBED) technique.

**Results**

The calculated phase mole fraction of the new alloy TT1 as a function of temperature is shown in Figure 1 (left). Only three types of precipitates, i.e., $M_{23}C_6$, MC, and Laves phase, exist in this alloy. The amount of MC is increased to be higher than that including M(C,N) in other developed RAFM steels, leading to the reduced amount of $M_{23}C_6$ in the alloy. Tensile testing results with yield strength and total elongation as a function of testing temperatures up to 800°C are shown in Figure 1 (right). The NIMS data of P92 tested using regular size specimens are included for comparison. Two N&T conditions with a slight difference in normalization temperature were conducted on alloy TT1. The yield strength of the new alloy is significantly higher than P92, e.g., ~290 and ~130 MPa greater than P92 at room temperature and 800°C, respectively. The total elongation of alloy TT1 is greater than 14%. According to the ASTM standard A335-11, the minimum elongation value of type SS-3 specimens is determined to be 10.95% by $E = 1.25t + 10.00$ for P92 miniature specimens with thickness $t = 0.76$ mm. Thus, the elongation of alloy TT1 is greater than the minimum elongation requirement of P92. Detailed microstructural characterization and thermal aging experiments of the samples of alloy TT1 are in progress.

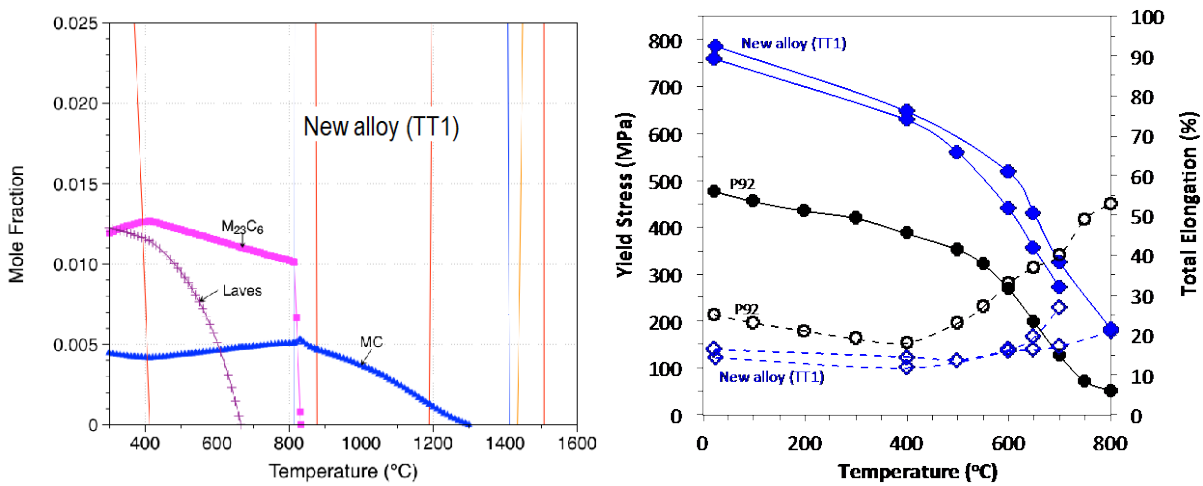


**Figure 1**. Temperature-dependent of calculated phase fraction (left) and tested yield strength and total elongation using type SS-3 specimens (right) of the newly developed alloy TT1. The NIMS data of P92 are included for comparison.

Four types of TMT conditions, involving the application of WR and tempering, have been developed on Grade 92 samples. Yield strength of the four TMT conditions, i.e., N+T+WR (C1), N+T+WR+T (C2), N+WR+T (F1), and N+WR+2T (F2), as a function of testing temperatures up to 650°C is shown in Figure 2. The WR after N&T (C1) noticeably increased strength, but the followed T (C2) significantly decreased strength, suggesting the occurrence of significant recovery. Although strength is decreased in the C2 condition, it is still noticeably higher than P92 as shown in Figure 1 (right). The application of WR between N and T (F1) exhibited strength between the C1 and C2 conditions. The application of a secondary tempering (F2) did not result in noticeable difference from the F1 condition. However, Charpy impact testing results indicated the lowest DBTT of the F2 (-43°C) and the highest DBTT of the C1 (35°C). Therefore, the F2 condition would be the best TMT in the four conditions, which will be applied to alloy TT1.

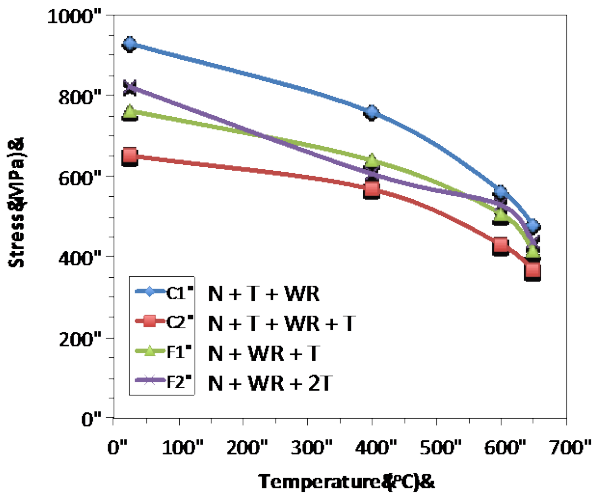


**Figure 2**. Effect of TMT on temperature-dependent yield strength of Grade 92.

The stability of TaC nanoprecipitates in ferritic steel under $Fe^{2+}$ ion irradiation to ~246 dpa at 500°C was characterized using scanning-transition electron microscopy (TEM/STEM). The left high-angle annular dark field (HAADF) image in Figure 3 was taken at ~1 μm from the TEM specimen surface, which corresponds to ~150 dpa according to SRIM (Stopping and Range of Ions in Matter) calculations [3] with input parameters recommended by Stoller *et al.* [4]. Compared to the right HAADF image taken from a non-irradiated zone (~2.5 μm from the TEM specimen surface), the size of the TaC particles (in white) was significantly reduced. Additionally, a few irradiation induced cavities, e.g., black spheres in the left HAADF image, were observed in the high dose irradiation samples; these were not observed in previous low dose irradiated samples. Statistical analysis of the results is in process.

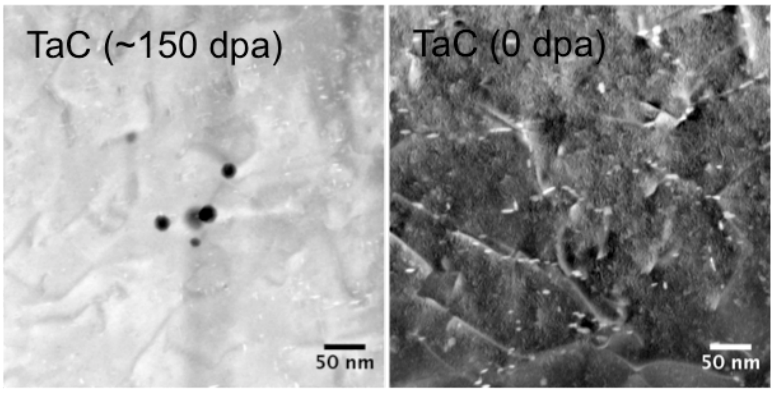


**Figure 3.** HAADF-STEM images of TaC nanoprecipitates in the ~150 dpa $Fe^{2+}$-irradiated (left) and non-irradiated (right) zones.

## REFERENCES

[1] L. Tan, Y. Kotah, L.L. Snead, Stability of the strengthening nanoprecipitates in reduced activation ferritic steels under $Fe^{2+}$ ion irradiation, J. Nucl. Mater. 445 (2014) 104-110.
[2] L. Tan, T.S. Byun, Y. Katoh, L.L. Snead, Stability of MX-type strengthening nanoprecipitates in ferritic steels under thermal aging, stress and ion irradiation, Acta Materialia 71 (2014) 11-19.
[3] The Stopping and Range of Ions in Matter (SRIM) (http://www.srim.org).
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