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Report on the Production by Mechanical Alloying of New Heats of 14YWT

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SUMMARY

This report provides a summary of the new heats of 14YWT produced by mechanical alloying. Compared to the past heats of 14YWT, which were based on the nominal composition of Fe-14Cr-3W-0.4Ti-0.3Nb + 0.3Y_2O_3 (wt. %), the three new heats of 14YWT contain a lower W level (1% vs. 3%) and various Ti levels of 0.2%, 0.3% and 0.4%. Powders with compositions of Fe-14Cr-1W-0.2Ti, Fe-14Cr-1W-0.3Ti and Fe-14Cr-1W-0.4Ti were produced by ATI Powder Metals via Ar gas atomization. The mechanical alloying procedure consisted of ball milling the powder, canning and degassing the milled powder followed by consolidation of the powder by hot extrusion. Powder consisting of 1 kg mass containing 0.3% Y_2O_3 powder was ball milled using the high kinetic energy ball mill for 40 h in Ar gas atmosphere. A total of 2 kg of powder from each of the three compositions of 14YWT was produced. The 3 batches of milled powder were added to 4 inch diameter cans that were fabricated from low carbon steel and sealed by electron beam welding. Each of the cans were degassed for 24 h at 300ºC under vacuum. For consolidation of the milled powder, the cans were solutionized at 850ºC for 2 h and then at 1000ºC for 2 h followed by extrusion through a rectangular shaped die to form the 14YWT billets. The 14YWT heat containing 0.4%Ti (14YW4T) was successfully extruded before a critical component on the extrusion press failed, thus preventing any further extrusions until it is repaired.

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1. INTRODUCTION

Advanced nuclear reactors such as Generation IV fission reactor concepts will require structural materials for fuel cladding and core components that possess attractive high-temperature mechanical properties and are tolerant to extreme irradiation environments consisting of high operating temperatures, applied stresses and neutron displacement damage levels. Structural materials that meet these requirements will offer many benefits to development of Generation IV reactor concepts based on improved economics, sustainability, reliability and safety compared to the current generation of water-cooled fission reactors. One class of advanced materials that show promise for achieving these requirements are Nanostructured Ferritic Alloys (NFA), which are advanced versions of oxide dispersion strengthened (ODS) ferritic alloys that were developed in the 1970's and 1980's for liquid metal cooled fast reactors.

The NFA 14YWT was developed starting in 2001 at ORNL for extreme neutron irradiation environments such as plasma facing components of fusion reactors and fuel cladding of fast fission reactors. The goals in development of 14YWT were to obtain ultra-fine grains plus a high concentration of nano-size Y-, Ti- and O-enriched nanoclusters (NC) for achieving high-strength and creep properties at elevated temperatures and providing high sink strength for trapping point defects to enhance recombination and for trapping transmutated He to form a high concentration of nano-size bubbles. Research and development (R&D) of NFA 14YWT has continued in the NTRD program since 2009 with goals on improving the mechanical alloying processing for scale up production, fabricating thin wall tubing and joining components such as thin tubes and plates. Fabricating and joining complex components such as thin wall fuel cladding for fast reactor concepts present significant technological challenges for NFA 14YWT due to the high strength properties and formation of microstructural textures that are detrimental to plastic deformation during fabrication. The long history of R&D of NFA 14YWT has resulted in great understanding of the relationship between processing, microstructure and mechanical properties. However, there have been no attempts to investigate the role of composition on these relationships. Therefore, the purpose of this research is to explore the effects of lowering the tungsten level and investigating different titanium levels on the microstructure and mechanical properties of NFA 14YWT. The goal of this research will be to determine if improvements in the balance of mechanical properties, such as strength versus ductility, high-temperature creep and fracture toughness, and radiation tolerance can be achieved for NFA 14YWT.

2. PROCESSING OF 14YWT HEATS

2.1. Powder

The nominal composition of 14YWT (benchmark) was Fe-14Cr-3W-0.4Ti + 0.3% Y_2O_3 (wt.%). For the new heats of 14YWT, it was decided to alter the tungsten (W) and titanium (Ti) levels to investigate their effects on the microstructure and mechancial properties compared to the benchmark 14YWT containing 3%W and 0.4%Ti. The W level was lowered to 1% partly due to the ineffectiveness of solid soluton strengthenging effects at high temperatures that 14YWT was developed for. Another reason was due to W favoring the formation of carbides that have complicated crystal structures, such as M_6C and $M_{23}C_6$ along with Cr, where M = Fe, Cr and/or W. Lowering the possibily to form these carbides should improve the fracture toughness of 14WYT since they typically have large blocky-shaped morphologies that can act as stress concentrators for crack formation. Three Ti levels were selected, one maintaining the 0.4%Ti level consistent with the benchmark 14YWT, while lower levels of 0.2%Ti and 0.3%Ti were chosen to investigate their effects on formation of the high concentration of nano-size Ti-Y-O oxide particles and the lower number density of larger Ti(CN) carbonitrides. The Ti(CN) particles are often inhomogeneously distributed in the microstructure and may play a role in fracture toughness properties.

The pre-alloyed powder of the three new heats of 14YWT with nominal compositions of Fe-14Cr-1W-0.2Ti, Fe-14Cr-1W-0.3Ti and Fe-14Cr-1W-0.4Ti was produced by ATI Powder Metals by Ar gas atomization. Twenty pounds (~9.07 kg) of powder was produced for each heat and sieved to a particle size range of 150 to 45 µm (-100/+325 mesh). The chemical analysis of the powders obtained by ATI Powder Metals is shown in Table 1. In this analysis, C was measured with LECO Compustion method, O and N were measured with LECO Fusion method and all other elements were measured by Inductive Coupled Plasma method. All composition specifications were achieved by ATI Powder Metals.

Table 1. Composition of the three heats of 14YWT powder.

Powder	Heat Designation	Composition (wt.%)						
		Fe	Cr	W	Ti	O	N	C
Fe - 14Cr - 1W - 0.2Ti	14YW2T	Bal.	13.80	1.02	0.14	0.011	0.002	0.010
Fe - 14Cr - 1W - 0.3Ti	14YW3T	Bal.	13.80	0.98	0.33	0.012	0.001	0.004
Fe - 14Cr - 1W - 0.4Ti	14YW4T	Bal.	13.80	1.03	0.41	0.010	0.001	0.005

2.2. Ball milling

The gas atomized powders of the three 14YWT heats were mixed with 0.3 wt.% Y_2O_3 powder that had a crystallite size range of 17-31 nm. The blended powder of 1 kg mass was ball milled using the high kinetic energy Zoz CM08 Simoloyer under Ar atmosphere for 40 h. Figure 2 shows the CM08 Simoloyer that was used for the ball milling runs. The ball milling run was conducted using 5 mm diameter low carbon steel milling media with a ball-to-powder ratio of 10:1. A total of 2 kg of powder was produced by ball milling for each of the three 14YWT heats and are shown in Figure 2. The heat designations of the powders after ball milling with 0.3 wt.% Y_2O_3 powder are shown in Table 1.

Figure 1. The high kinetic energy Zoz CM08 Simoloyer.

2.3. Extrusion

The procedure for extruding the ball milled powder of the three 14YWT heats consisted of fabricating cans for holding the ball milled powder, degassing the can packed with powder and hot extrusion. The cans were fabricated from low carbon steel and had dimensions of 3.9 in. diameter and 5.5 in. long. A lid to the cans with a 0.25 in. (6.35 mm) diameter opening at the center was fabricated from low carbon steel. Figure 2 shows the fabricated cans and lids. The ball milled powder was packed in each can and the lid was welded to the can. A 0.25 in. diameter stainless steel tube with fixture for connecting to the vacuum system was welded to the lid. The can was degassed under vacuum for 24 h at 300ºC. The stainless steel tube was crimped following the vacuum degassing.

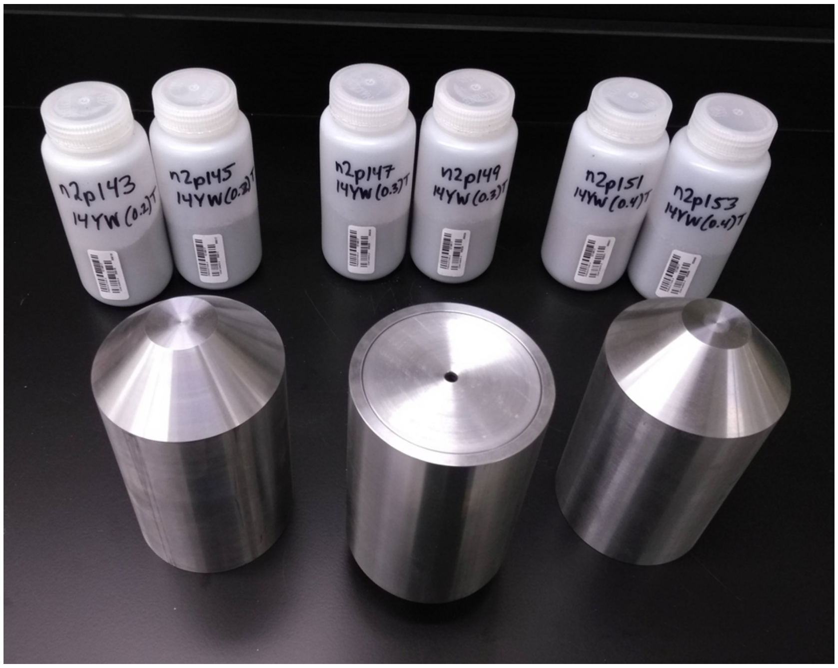

Figure 2. Image showing the 3.9 in. diameter x 5.5. in. long cans and lids fabricated from mild steel and the plastic bottles containing the ball milled powder of the three new 14YWT heats.

The Watson-Stillman press, which has a capacity of 1,250 tons (~1.13 x 10^6 kg) was used to extrude the sealed cans. The cans were solutionized in an open-air furnace at 850ºC for 2 h and then at 1000ºC for 2 h followed by extrusion through a rectangular shaped die that had an opening of 2.5 in. (6.35 cm) wide x 1.25 in. (3.175 cm) high. Figure 3 shows the extrusion press (Fig. 3a) and the extruded bar of 14YW4T (Fig. 3b). After cutting the nose and tail sections off of the extruded bar to reveal the enclosed ODS section, the final length of the extruded 14YWT bar was ~9 inches. Figure 4 shows the extruded bar and a ruler for scale. The remaining 2 heats (14YW2T and 14YW3T) will be extruded following the repair of the extrusion press, which suffered a failure after extrusion of 14YW4T.


(a)
(b)

Figure 3. Images showing (a) the Watson-Stillman extrusion press and (b) the rectangular shaped bar of 14YW4T after extrusion.

3. FUTURE PLANS

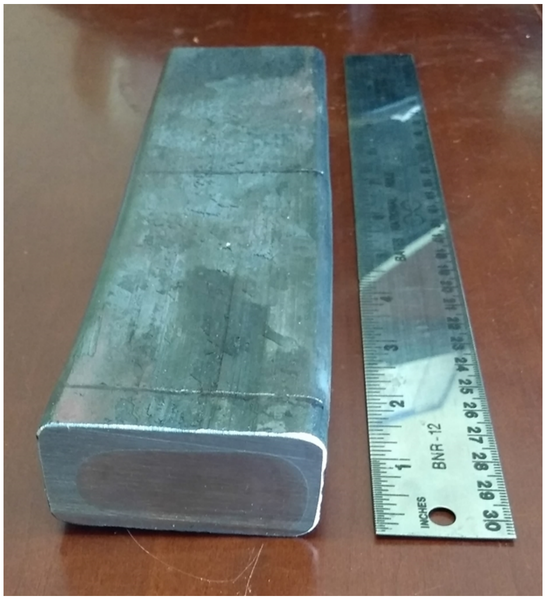

Figure 4. The extruded bar of 14YW4T after cutting the nose and tail sections off.

The two remaining sealed cans will be extruded using the same conditions as 14YW4T once the extrusion press is repaired and functioning. The extruded bars will be cut near the center of the bar. A sample of 0.25 inches thick will be cut from one end of the extruded bars for preparing samples for chemical analysis and initiation of the microstructure characterization studies. This will be done by mounting two samples cut with orientations parallel and normal to the extrusion direction followed by polishing with a final polished using colloidal silica. The microstructures of the polished samples will be examined by Scanning Electron Microscopy (SEM) using backscattered electron (BSE) imaging and electron backscattered diffraction (EBSD) for texture mapping. Tensile specimens will be fabricated from the extruded bar and tested at temperatures ranging from room temperature (25ºC) to 800ºC to assess the temperature dependent strength and ductility properties of the three new 14YWT heats for investigating the effects of lower 1%W and various 0.2%Ti, 0.3%Ti and 0.4%Ti levels for comparison.

4. SUMMARY

Three new heats of 14YWT were developed to investigate the effects of lowering the W level and different Ti levels on the microstructure and mechanical properties for comparing with past heats of 14YWT. The goal of this research will be to determine if improvements in the balance of mechanical properties, such as strength versus ductility, high-temperature creep and fracture toughness, and radiation tolerance can be achieved with the new heats of 14YWT. Compared to past 14YWT heats that had a composition of Fe-14Cr-3W-0.4Ti + 0.3Y_2O_3, the three new 14YWT heats contained 1%W and either 0.2%Ti, 0.3%Ti or 0.4%Ti. Powders with compositions of Fe-14Cr-1W-0.2Ti, Fe-14Cr-1W-0.3Ti and Fe-14Cr-1%W-0.4Ti were produced by ATI Powder Metals via Ar gas atomization. The mechanical alloying procedure consisted of ball milling the powder, canning and degassing the milled powder followed by consolidation of the powder by hot extrusion. Powder consisting of 1 kg mass containing 0.3% Y_2O_3 powder was ball milled using the high kinetic energy CM08 Simoloyer for 40 h in Ar gas atmosphere. A total of 2 kg of powder from each of the three compositions of 14YWT was produced. The 3 batches of milled powder were added to 4 in. diameter cans that were fabricated from low carbon steel and sealed by electron beam welding. Each of the cans were degassed for 24 h at 300ºC under vacuum. For consolidation of the milled powder, the cans were solutionized in an open-air furnace at 850ºC for 2 h and then at 1000ºC for 2 h followed by extrusion through a rectangular shaped die to form the 14YWT billets. The 14YWT heat containing 0.4%Ti (14YW4T) was successfully extruded before a critical component on the extrusion press failed, thus preventing any further extrusions until it is repaired.