2.3 Specimen Preparation and characterization of 14YWT (SM12 heat) for the JP-30/31 HFIR Experiments — D. T. Hoelzer, E. T. Manneschmidt and M. A. Sokolov (Oak Ridge National Laboratory)

OBJECTIVE

To fabricate specimens from the advanced oxide dispersion strengthened (ODS) 14YWT (SM12 heat) ferritic alloy for the JP-30 and JP-31 HFIR target irradiation experiments and conduct the reference characterization of the microstructure and mechanical properties of three SM12 heats that were produced with different extrusion temperatures and rolling conditions to compare with PIE results that will be obtained in the future.

SUMMARY

Three heats of the advanced ODS 14YWT-SM12 ferritic alloy were produced with different extrusion temperatures and plates of each heat were fabricated with two different rolling conditions. The 14YWT-SM12 was a "clean" heat that was produced with improved mechanical alloying conditions to achieve low C (128 wppm) and N (117 wppm) levels and elevated O (1184 wppm) level. Plates of three SM12 heats were obtained by extruding at 850ºC, 1000ºC and 1150ºC temperatures followed by rolling at 1000ºC to 50% reduction in thickness in either the parallel (PR) or cross (CR) directions with reference to the extrusion direction. Specimens were fabricated from four plates of the SM12 heat with different thermal-mechanical treatments (TMT) and supplied for assembly of the JP30/31 capsules. The reference characterization of the microstructure and mechanical properties of the three SM12 heats was initiated. Results obtained from tensile tests at 25ºC showed slightly lower strengths, but significantly higher uniform (>9%) and total elongations in all the 14YWT-SM12 heats compared to results obtained from previous 14YWT heats. The general microstructural characteristics observed by SEM analysis showed that slightly larger grains with less uniformity in grain size had formed in the SM12 heats compared to previous 14YWT heats. The observed differences in the mechanical properties and grain structures of the SM12 heats compared to previous 14YWT heats were most likely due to lowering the O, C and N levels during ball milling.

PROGRESS AND STATUS

Introduction

The new target irradiation experiment (JP-30/31) was developed within the framework of the U.S.DOE-JAEA collaboration on fusion materials to investigate the effects of neutron irradiation at different temperatures on the microstructure stability and mechanical properties of reduced-activation ferritic/martensitic (RAFM) steels and the advanced dispersion strengthened ferritic alloys and FM steels [1]. The JP-30/31 experiment was modified from the JP-28 and JP-29 experiments by designing the capsules to increase the neutron irradiation temperature for one of the sub-capsules from 500ºC (JP-28/29) to 650ºC (JP-30/31) while maintaining similar irradiation temperatures of 300ºC and 400ºC for the other two sub-capsules [2]. The estimated neutron fluence of the JP-30/31 experiment is ~20 dpa. One of the primary reasons for raising the irradiation temperature to 650ºC was to investigate the effects of irradiation on the microstructure stability and mechanical properties of advanced dispersion strengthened materials, such as oxide dispersion strengthened (ODS) alloys that are being developed specifically for nuclear energy components that required higher operating temperatures.

The U.S. Fusion Materials program selected the advanced ODS 14YWT ferritic alloy for the JP-30/31 experiment since a fairly large background on processing, microstructure analysis and mechanical properties characterization have been obtained from studies of 14YWT heats produced in the past. For the JP-30/31 experiment, the 14YWT (SM12 heat) was used for preparation of specimens. The 14YWT-SM12 was produced in the Core Materials Technologies project of the Fuel Cycle Research and Development (FCRD) NE program. The primary goal of the project was to optimize the processing conditions for scale-up production of the ~55 kg heat of 14YWT. A key processing factor investigated in this project was to improve the ball milling conditions to minimize the C and N contents while maintaining a high O content, which is required for forming the high concentration of nano-scale oxide particles that is characteristic of 14YWT heats. The purpose of this report is to cover the processing conditions used for producing three 14YWT-SM12 heats, the type and number of specimens that were prepared for the JP30/31 HFIR experiment and the preliminary results obtained from the reference characterization of the microstructures and mechanical properties of the 14YWT-SM12 heats.

Processing of the 14YWT-SM12 Heats

The 14YWT-SM12 heat was produced by mechanical alloying (MA) using gas atomized powder produced by Special Metals with nominal composition of Fe-14Cr-3W-0.4Ti (Fe pre-alloyed) and nano-size (17-31 nm) Y_2O_3 powder produced by Nanophase, Inc. The blended powder consisting of 99.7% Fe pre-alloyed and 0.3% Y_2O_3 powder (wt. %) was ball milled using the Zoz CM08 high energy attritor mill for 40 h in an Ar atmosphere. Three ball milling runs consisting of 1.0 kg of blended powder each were performed, which generated 3.0 kg of ball milled powder. The new ball milling conditions developed in the FCRD program resulted in significantly lower O, C and N levels in the SM12 heat compared to previous 14YWT heats such as the SM11 heat as shown in Table 1. In the table, the amount of O that was picked up during ball milling was determined by subtracting both the O level in the atomized powder (~150 wppm) and the O level from the Y_2O_3 powder (683 wppm) from that of the ball milled powder. The results indicated that the O, C and N levels picked up during ball milling were significantly lower in the SM12 powder compared to the SM11 powder.

For consolidation of the 3.0 kg of ball milled powder by extrusion, the powder was first separated into 4 equal batches of 750 g. Three of the batches of ball milled powder were sealed in 2.9 inch diameter mild steel cans and degassed at 400ºC in vacuum before being hermetically sealed. The sealed cans were first annealed at 850ºC for 1 h. The cans were then annealed for 1 at three different temperatures corresponding to the extrusion temperatures. One can was extruded at 1150ºC (SM12a heat), one was extruded at 1000ºC (SM12c heat) and the last one was extruded at 850ºC (SM12d heat). The cans were extruded through a rectangular shaped die to form bars. Extruding the cans at different temperatures was intended

Table 1. Comparison of the O, C and N levels of the SM11 and SM12 heats of 14YWT.

Element	14YWT-SM11			14YWT-SM12		
(wppm)	Atomized	Milled	Pickup	Atomized	Milled	Pickup
O	150	2590	1802	150	1184	396
C	100	346	246	100	128	28
N	50	1911	1861	50	117	67

to alter both the grain size and oxide particle dispersion between the 14YWT-SM12 heats and to study which condition may provide the best combination of mechanical properties before and after irradiation.

After extrusion, the bars of the three 14YWT-SM12 heats were annealed at 1000ºC for 1 h in vacuum and then were cut into 3 sections of equal lengths for rolling. The rolling procedure used for each heat was performed by rolling one bar parallel (PR) and another bar normal, or cross (CR), to the extrusion direction to form plates as shown in Figure 1. The rolling of the bars into plates was conducted at 1000ºC with a reduction in thickness of ~50%. The cross rolling (CR) was performed to investigate whether the anisotropy in the mechanical properties that is typical of mechanically alloyed ODS alloys could be minimized.

Figure 1. Digital image of SM12a showing the three cut sections of the extruded bar in the as extruded condition (center), hot rolled parallel to the extrusion axis (right) and hot rolled normal (cross) to the extrusion axis (left).

Preparation of Specimens for the JP-30/31 HFIR Experiment

Specimens for the JP30/31 HFIR experiment were fabricated from the three PR plates of each heat (SM12a, SM12c and SM12d) and from the CR plate of the SM12d heat. There were six types of specimens that were prepared. The specimens consisted of miniature tensile (SSJ3; 5 x 1.4 x 0.75 mm gage), 5 notch fracture toughness bend bar (M5PCCVN; 54 x 3.3 x 1.65 mm), disk compact tension fracture toughness (DCT; ~ø12.5 x 0.2 mm), tuning fork type specimens for atom probe analysis (APFIM; 16 x 1.14 x 0.5 mm), TEM disks (ø3 x 0.25 mm) and miniature multi-purpose coupons (MMPC; 16 x 1.15 x 1 mm). The list showing the type and number of specimens that were prepared from the four plates is shown in Table 2.

Table 2. The specimens fabricated from the 14YWT-SM12 heats for the JP-30/31 experiment.

Capsule	14YWT Heat	Extrusion Temperature	Specimen Type	Quantity
300ºC	SM12a-PR	1150ºC	APFIM	2
			TEM	4
	SM12c-PR	1000ºC	APFIM	2
			TEM	4
	SM12d-PR	850ºC	APFIM	2
			TEM	4
			SS-J3	4
			DCT	6
400ºC	SM12a-PR	1150ºC	APFIM	2
			TEM	4
	SM12c-PR	1000ºC	TEM	4
	SM12d-PR	850ºC	APFIM	2
			TEM	4
650ºC	SM12a-PR	1150ºC	APFIM	2
			TEM	4
			SS-J3	6
	SM12c-PR	1000ºC	APFIM	2
			TEM	4
			SS-J3	6
	SM12d-PR	850ºC	APFIM	2
			TEM	4
			SS-J3	6
			DCT	9
	SM12d-CR	850ºC	M5PCVVN	4
			SS-J3	4
			MMPC	5

The assignment of the specimens for the sub-capsules that will be neutron irradiated at 300ºC, 400ºC and 650ºC is also shown in Table 2. The test matrix emphasized that PIE will consist of microstructural analysis at each irradiation temperature and mechanical properties examination only at the low (300ºC) and high (650ºC) irradiation temperatures. Both tensile and fracture toughness properties will be measured at the two irradiation temperatures.

Reference Characterization Study of the 14YWT-SM12 Heats

The detailed characterization of the microstructure and mechanical properties of the 14YWT-SM12 heats was performed to allow comparison with irradiated specimens from the JP30/31

experiment and also to investigate the role that the different processing and fabrication conditions had on the grain structure and nano-scale oxide particle dispersion that formed in the heats and to determine how differences in the microstructures affected the mechanical properties. The reference characterization study will focus on microstructure analysis using SEM, TEM and possibly Atom Probe and on mechanical properties characterization using hardness, tensile and fracture toughness tests.

Samples obtained from the plates of SM12a (1150ºC), SM12c (1000ºC), and SM12d (850ºC) in the PR condition and SM12d (850ºC) in the CR condition were mounted and polished for Vickers Hardness measurements and microstructural analysis by SEM. The Vickers Hardness Number (VHN) obtained from the four samples is shown in Figure 2. The results showed the expected trend of an increase in hardness with decrease in extrusion temperature, but the range between the lowest and highest hardness value was relatively small (~42 VHN, or ~10%). The largest difference in hardness occurred between the 850ºC heats (SM12d PR and CR) and the 1000ºC heat (SM12c PR). The microstructure of the samples was characterized with the SEM using back scattered electrons (BSE) to improve imaging ultra-small grains common to 14YWT heats. The BSE images observed in Figure 3 were obtained at low magnification to show the uniformity of grain sizes that were present in the heats. As shown in this figure, all of the SM12 heats showed grain sizes that were not very uniform compared to previous 14YWT heats. At the higher magnification shown in Figure 4, the BSE images revealed that the

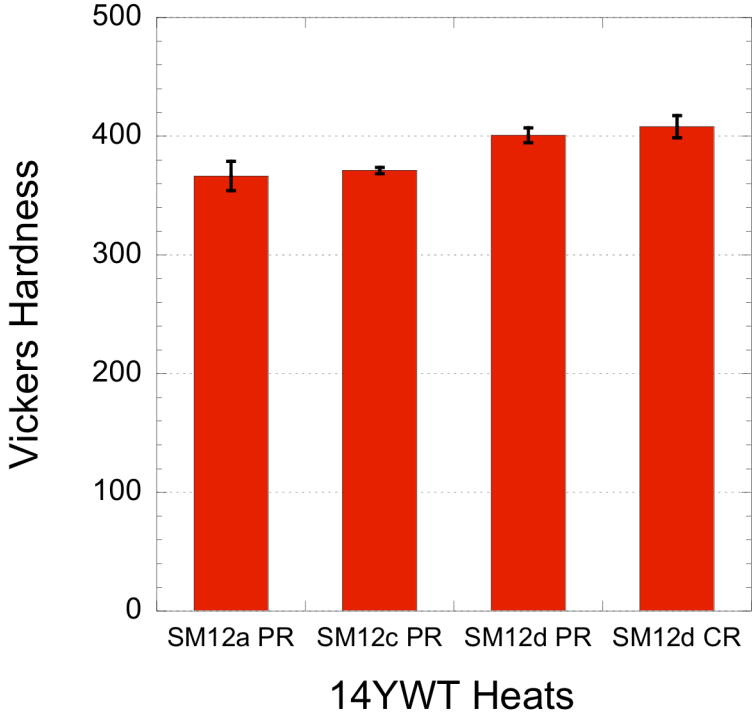

Figure 2. Comparison of Vickers Hardness for the 14YWT-SM12 heats.

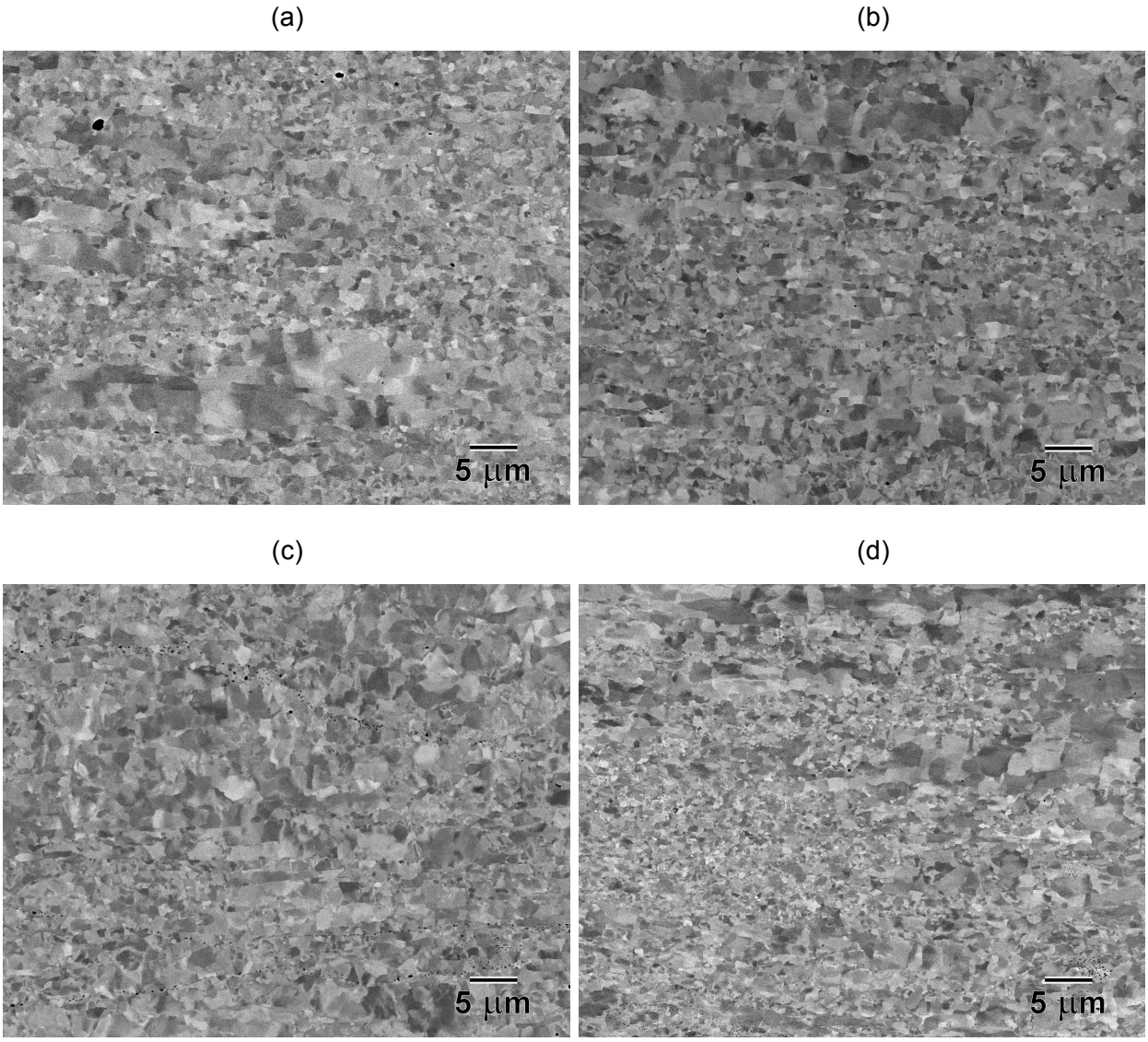

Figure 3. SEM BSE images at a relatively low magnification showing the variation in grain size observed in the microstructures of the (a) SM12a PR, (b) SM12c PR, (c) SM12d PR and (d) SM12d CR heats.

grain were also slightly larger in size compared to previous 14YWT heats. In addition, small dark imaging particles were observed (Figures 4c and 4d) usually distributed in clusters along interfaces that were most likely prior surfaces of the ball milled powder. Interestingly, the comparison of both sets of BSE images shown in Figures 3 and 4 indicated that the different extrusion temperatures and rolling conditions had very little effect on the microstructures since they were very similar in the SM12 heats.

Tensile tests were performed on SS-J3 specimens of the SM12 heats at room temperature (25ºC) using a strain rate of 1 x 10^{-3} s^{-1}. The stress strain curves shown in Figure 5 revealed slightly lower strengths, but significantly higher uniform and total elongations in all the

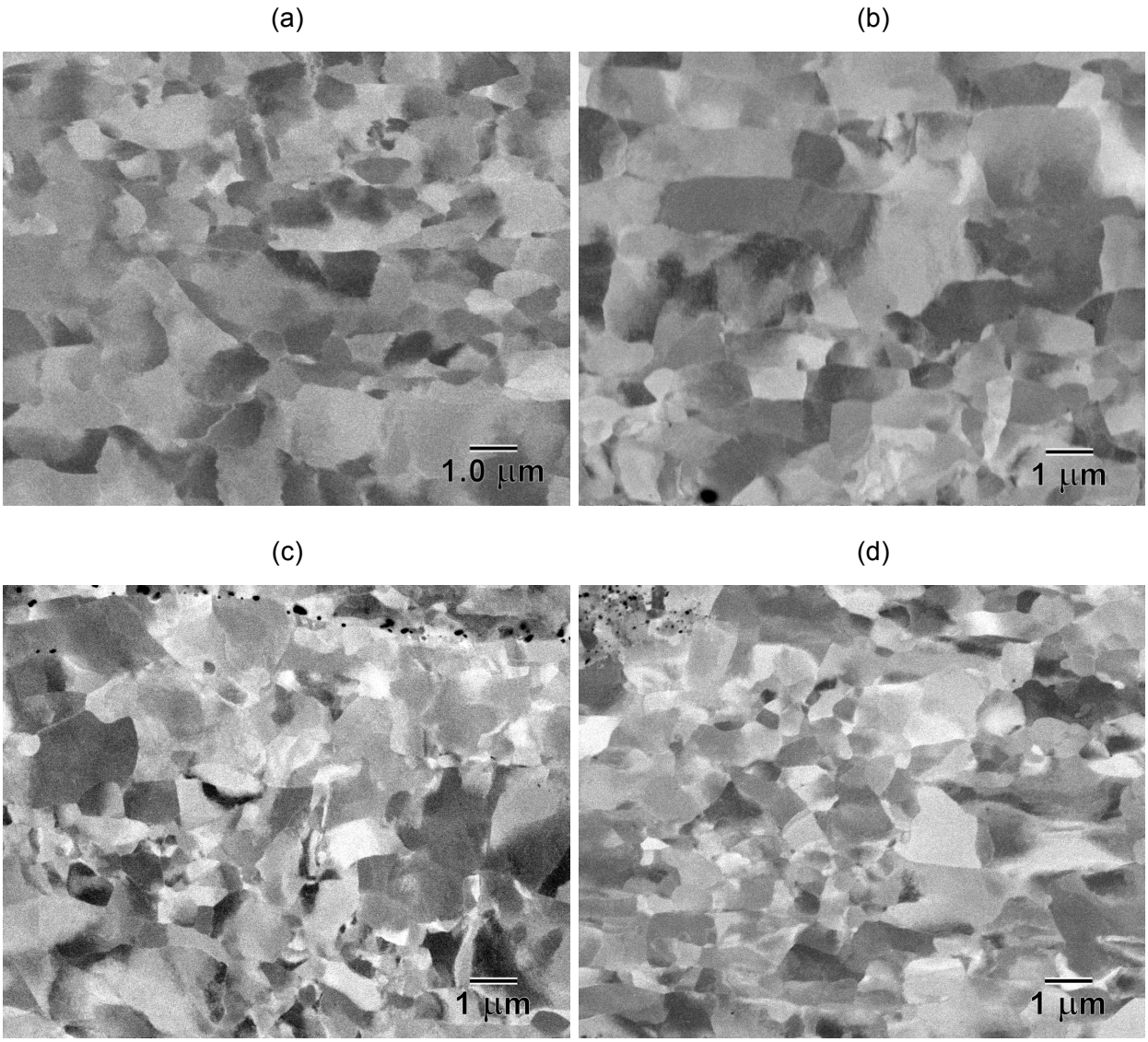

Figure 4. SEM BSE images at high magnification showing the grain size observed in the microstructures of the (a) SM12a PR, (b) SM12c PR, (c) SM12d PR and (d) SM12d CR heats.

14YWT-SM12 heats compared to results obtained from previous 14YWT heats. All of the SM12 heats possessed uniform elongations that were greater than 9%, which is significantly better than the 1 to 2% uniform elongation typically observed in previous 14YWT heats. The yield and ultimate tensile stresses and uniform and total elongations measured from the stress-strain curves of the SM12 heats are shown in the plot in Figure 6. Interestingly, the measured values showed essentially no differences between the heats, indicating that differences in the extrusion temperature and rolling conditions had very little effect on the tensile deformation behavior. The

yield stress and ultimate tensile strengths of the SM12 heats were all near ~1.0 GPa and ~1.2 GPa, respectively. In a similar manner, the uniform and total elongations were nearly constant for each SM12 heat and were ~9-10% and ~21-24%, respectively.

The combination of relatively high strength with much improved ductility properties was most likely due to the lower C and N levels that were achieved in producing the SM12 heat. Furthermore, the lower strength and higher ductility of the SM12 heats compared to previous 14YWT heats may also be due to the larger grain sizes that were observed. However, further microstructural characterization using TEM analysis to investigate the nano-scale oxide particle dispersions in the SM12 heats is necessary to obtain a better understanding of the relationship between processing, mechanical properties and microstructure of the 14YWT-SM12 heats.

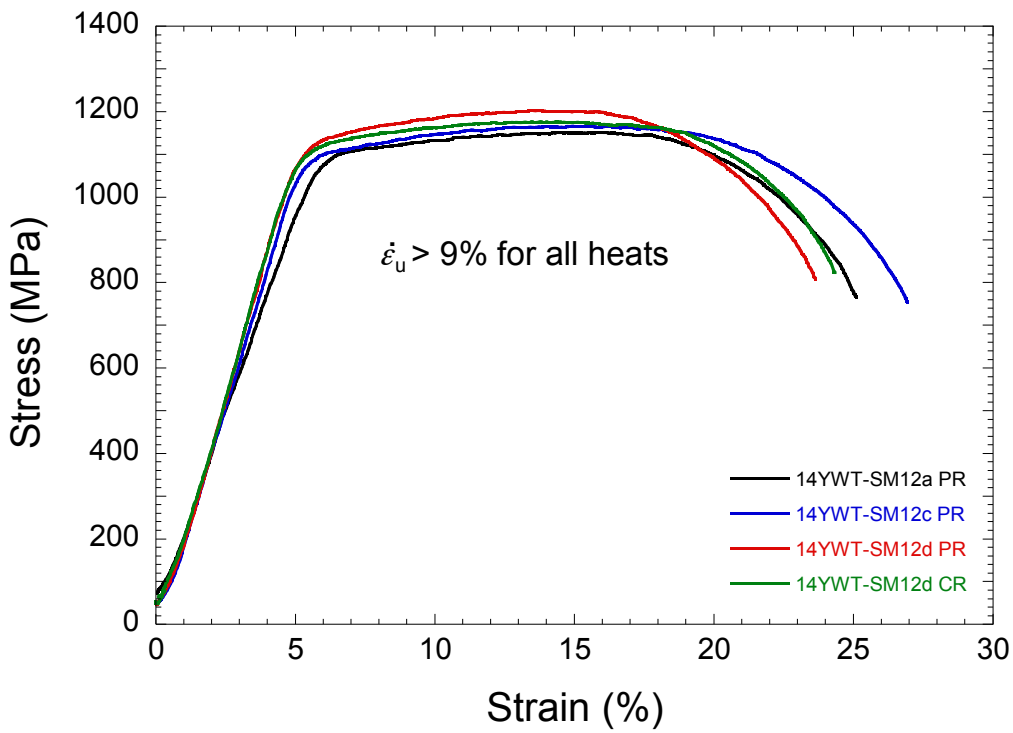

Figure 5. Stress-strain curves of the SM12a PR, SM12c PR, SM12d PR and SM12d CR heats obtained from tensile tests at 25ºC using a strain rate of 1 x 10^{-3} s^{-1}.

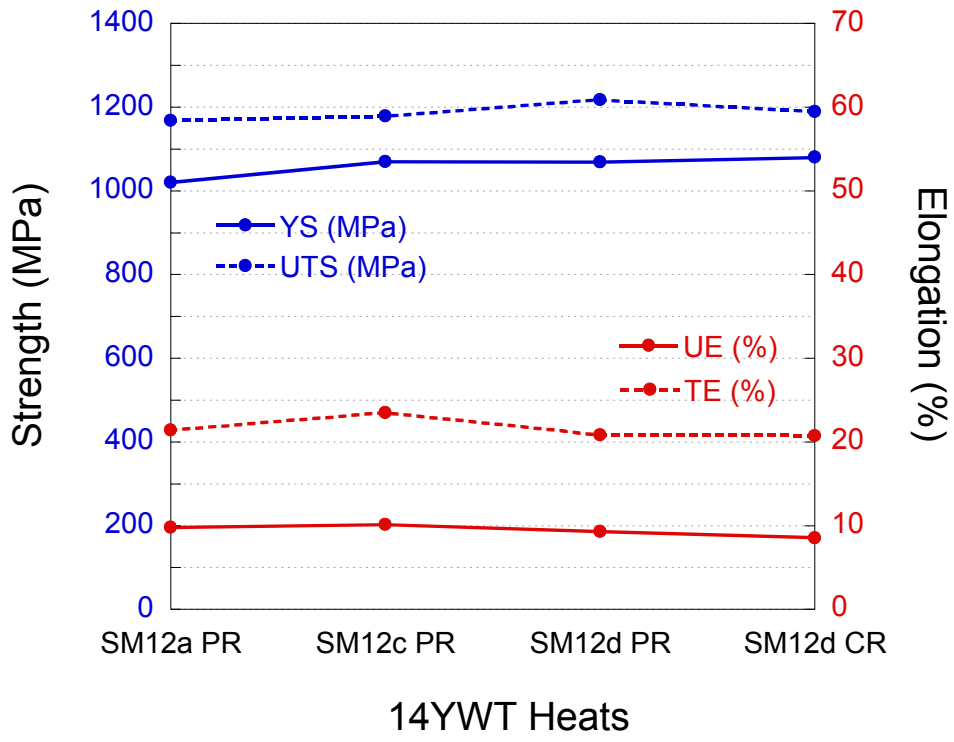

Figure 6. Comparison of the tensile properties of the SM12a PR, SM12c PR, SM12d PR and SM12d CR heats obtained from tensile tests at 25ºC using a strain rate of 1 x 10^{-3} s^{-1}.

SUMMARY

Three heats of the advanced ODS 14YWT-SM12 ferritic alloy were produced by extrusion at 850ºC, 1000ºC and 1150ºC. Improved mechanical processing conditions led to low C and N levels and elevated O level for 14YWT-SM12. Plates of the three SM12 heats were fabricated by rolling at 1000ºC to 50% reduction in thickness in the parallel (PR) and cross (CR) rolling directions with reference to the extrusion direction. Specimens were fabricated from four plates of the SM12 heats and supplied for assembly of the JP30/31 capsules for the JP-30/31 HFIR irradiation experiments. The reference characterization of the microstructure and mechanical properties of the three SM12 heats was initiated. Results obtained from tensile tests at 25ºC showed slightly lower strengths, but significantly higher uniform (>9%) and total elongations in all the 14YWT-SM12 heats compared to results obtained from previous 14YWT heats. The general microstructural characteristics observed by SEM analysis showed that slightly larger grains with less uniformity in grain size had formed in the SM12 heats compared to previous 14YWT heats. The observed differences in the mechanical properties and grain structures of the SM12 heats compared to previous 14YWT heats were most likely due to lowering the O, C and N levels during ball milling.

FUTURE WORK

Detailed characterization of the microstructure and mechanical properties of the 14YWT-SM12 heats will continue. TEM specimens will be jet-polished soon and examined. Plates of the SM12a PR, SM12c PR and SM12d PR will be submitted to the machine shop for fabricating a number of SS-J3 tensile and DCT fracture toughness specimens for further mechanical properties testing. The tensile tests will emphasize elevated temperatures while the fracture toughness tests will focus on determining the fracture toughness transition temperature and possibly the fracture toughness at elevated temperatures. The goal is to have a sufficiently large number of SS-J3 and DCT specimens prepared so that several specimens can heat treated at similar temperatures for the same period of time that specimens in the JP-30/31 experiment will be experience. Comparison of the microstructure and mechanical properties that will be obtained from the annealed and irradiated specimens of the SM12 heats will provide valuable information about the effect of time at temperature so that a more accurate assessment of the effect that neutron irradiation may have on changes in the microstructures and mechanical properties can be determined.

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