2.2 A NEW SCANDIUM-BASED NANOSTRUCTURED FERRITIC ALLOY – T. Stan, S. Pun, N. J. Cunningham, Y. Wu and G. R. Odette (University of California Santa Barbara)

OBJECTIVE

The objective of this work is to synthesize, characterize and, eventually, determine the thermal stability of a new type of nanostructured ferritic alloy (NFA) with much higher solubility Sc replacing the basically insoluble Y, potentially opening a melt processing path.

SUMMARY

A new Sc-based nanostructured ferritic alloy (NFA) was processed by ball milling gas atomized Fe alloy and Sc_2O_3 powders. Replacing Y with Sc was motivated by the fact that the latter has a much higher solubility in Fe compared to Y, possibly providing a melt processing path for producing Sc-based NFA. The milled powders were subject to four heat treatments: aging at both 850°C; aging at 1000°C; consolidation by spark plasma sintering (SPS) at a peak temperature of 1100°C; and SPS consolidation followed by annealing at 850°C for 3 hrs. Electron backscatter diffraction (EBSD) measurements showed fine grain sizes in both the 850°C and 1000°C aged powder conditions. Transmission electron microscopy (TEM) and atom probe tomography (APT) show that similar populations of $Sc_2Ti_2O_7$ complex oxide nano-features (NFs) also form at both aging temperatures. The microhardness was high for three conditions, and similar to that typically observed in Y-based NFA. However, the microhardness was much lower in the 1000°C powder aged condition. The reason for the low hardness is not yet understood.

BACKGROUND

Nanostructured Ferritic Alloys (NFAs) are Fe-Cr based ferritic stainless steels that traditionally contain an ultrahigh density of Y-Ti-O nanofeatures (NFs). NFAs have high tensile, creep and fatigue strengths, unique thermal stability and are irradiation tolerant [1-3]. The major thrust of this research is to explore a new NFA variant by replacing Y with Sc. Both Y and Sc are group III elements with similar chemical properties. However Sc is a much smaller atom than Y and has an enormously higher solubility with a maximum of 3 at% at 1280°C [4]. Thus, in principle, it should be possible to produce Sc-based NFA by more conventional melt processing paths. However, it is noted that that due to current high cost of Sc, this would be too expensive.

PROGRESS AND STATUS

Experimental Methods

An atomized Fe-based alloyed powder ball milled with Sc_2O_3 powders for 10 hours in a SPEX Mill 8000M, using 6 and 8 mm diameter balls, yielding a nominal powder composition of (in wt%) 14Cr, 3W, 0.4 Ti, 0.18Sc, bal Fe. Approximately 20 g batches of the milled powders were aged for 3 hours at 850°C and 1000°C. A third 20 g batch was consolidated by spark plasma sintering (SPS) under vacuum, using the conditions shown in Figure 1. The red curve is the temperature, the green curve is the applied pressure, held at 75 MPa, and the blue curve is the actuator displacement. The consolidated pellet was also subsequently annealed for 3 h at 850°C.

Microhardness data on the 850°C and 1000°C conditions was obtained from mounted powder samples with a LECO M-400A semi-automated indenter using a 500 g load. The microhardness was measured

directly on polished disks for both the 1100°C as-SPS and SPS plus annealed conditions. A FEI Quanta 400F field-emission environmental scanning electron microscope (SEM) with an electron backscatter diffraction (EBSD) detector was used to obtain grain size and crystallography information. EBSD was carried out on the 850°C and 1000°C annealed powders that were mounted in Bakelite and polished by first using a series of sandpapers down to 1200 grit, then 6 µm and 0.5 µm diamond powder on a fast-wheel, and finally with 0.02 µm colloidal silica. A FEI Helios focused ion beam (FIB) tool was used to micro-machine <30 nm thick electron transparent lift-outs for transmission electron microscopy (TEM). A 300 keV FEI Titan was used to obtain TEM and energy dispersive x-ray spectroscopy (EDS) information. Atom probe tomography (APT) was performed on an IMAGO LEAP 3000X HR microscope.

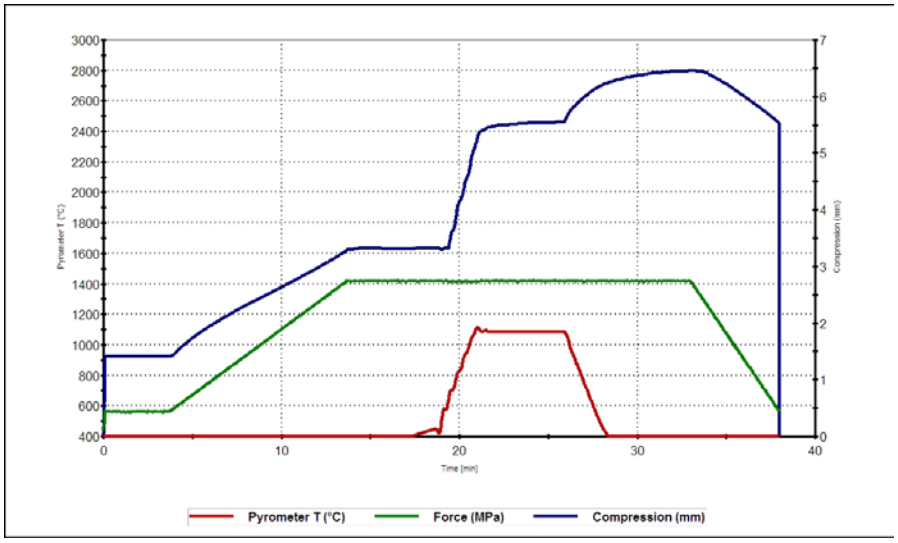

Figure 1. The SPS processing parameters used to consolidate the alloyed powder.

Results and Discussion

The microhardness data versus processing temperature for the Sc-based NFAs (blue) compared to typical values for Y-based NFA (red) are shown in Figure 2. Overall, the Y-based and Sc-based NFAs have similar hardness values. Surprisingly, the hardness of the 1000°C annealed Sc-NFA was lower than both of the 850°C anneal and the 1100°C SPS samples. This difference is not understood, but may be due to an artifact of measuring the hardness of the powders.

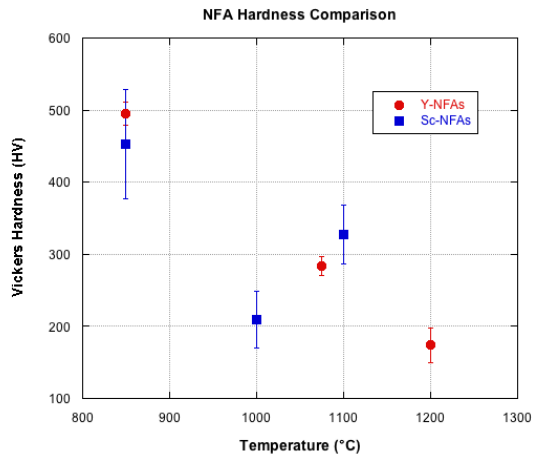

Figure 2. Microhardness of the Sc-NFAs (blue squares) and Y-NFAs (red diamonds).

Figure 3 shows EBSD pole figure maps. Figures 3a and 3b show the out of plane orientations of the 850°C and 1000°C aged powders, respectively. Red, green, and blue grains have out of plane orientations close to {100}, {110}, and {111}, respectively. The black/grey streaks in Figure 3b are areas where the grain orientation could not be determined. Pole figures (not shown) do not indicate crystallographic texturing in either case and both contain similar bimodal distributions of grain sizes, that range from <0.2 µm (fine) to >2 µm (coarser). Most of the larger grains were not uniformly colored which implies some plastic deformation. Grain size is not be the reason for the surprisingly low hardness 1000°C annealed condition.

Bright field TEM images for the 850°C and 1000°C powders are shown in Figures 4a and 4b, respectively. The dark features are precipitates that span a range of sizes. The average precipitate diameter in the 850°C and 1000°C samples is 3.3 nm and 2.9 nm, respectively. The number densities were also similar for the 850°C and 1000°C aged powders at 8.5×10^{22} m^{-3} (850°C) and 7.5×10^{22} m^{-3} (1000°C). APT measurements, illustrated in Figure 5, on the 850°C aged powders showed a higher density ≈ 2.6×10^{22} m^{-3} of somewhat smaller ≈ 2.1 nm precipitates.

Table 1 compares the precipitate sizes found in the Sc-based versus Y-based NFAs in various conditions and using various techniques (TEM, APT and SANS). The precipitates in the Sc-NFAs are similar in size and number density to those in Y-NFAs. EDS was used to measure the compositional information of the precipitates as summarized in Table 2 for the 850°C and 1000°C aged powder conditions. The precipitate diameters ranged from 7 nm to 52 nm in the 850°C aged powder condition with Sc/Ti ratios ranging from 0.5 to 1.2, averaging 0.72 ± 0.21. The corresponding (Sc + Ti)/O ratio ranged from 0.10 to 0.32 averaging 0.15 ± 0.37. There was no clear effect between precipitate size and Sc/Ti ratio and the (Sc + Ti)/O ratio. The precipitates in the 1000°C aged condition ranged from 5 nm to 40 nm, with similar Sc/Ti ratios ranging from 0.26 to 1.34, averaging 0.83 ± 0.39. The corresponding (Sc + Ti)/O ratio ranged from 0.08 to 0.24 averaging 0.17 ± 0.05. However in the 1000°C case, the smaller precipitates have larger Sc/Ti ratios. In both cases, the precipitates also contained trace amounts of W and Cr (not shown). The corresponding compositions found in APT characterization of the 850°C aged powder were Sc/Ti ≈ 0.83 and (Sc + Ti)/O ≈ 0.88. The APT and EDS Sc/Ti are in good agreement. The APT (Sc + Ti)/O are lower than found by EDS. However this is consistent with previous results on Y-based NFA. Overall the precipitates in the

annealed Sc-alloys appear to be Sc-Ti-O complex oxides, perhaps $Sc_2Ti_2O_7$, and the deviations in the nominal O are due to various limitations and artifacts in the techniques.

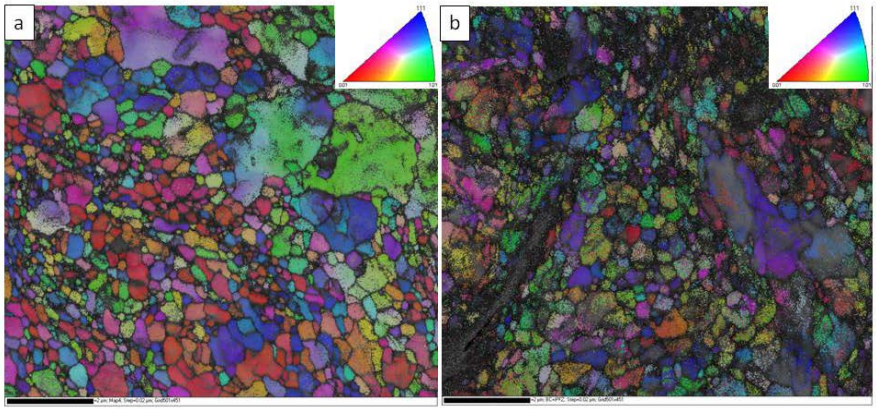

Figure 3. EBSD pole figure maps showing grain size from 850°C annealed powder (a) and from 1000°C annealed powder (b).

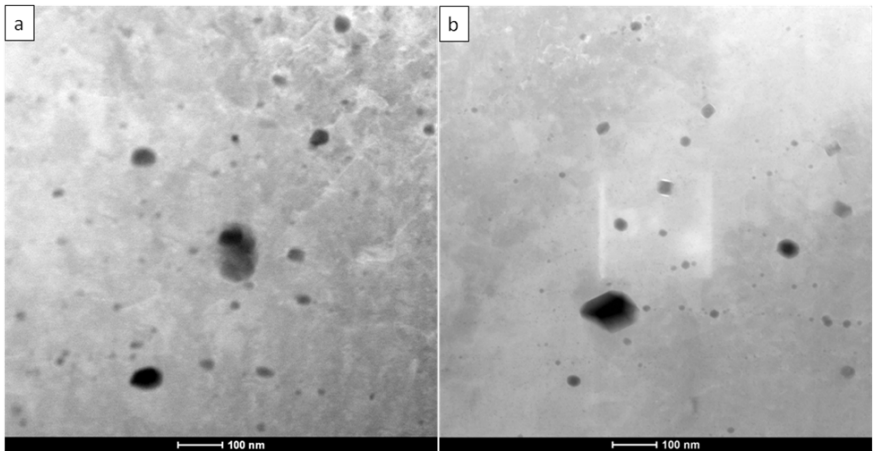

Figure 4. TEM micrographs for powders aged at 850°C (a) and 1000°C (b) for 3 hrs.

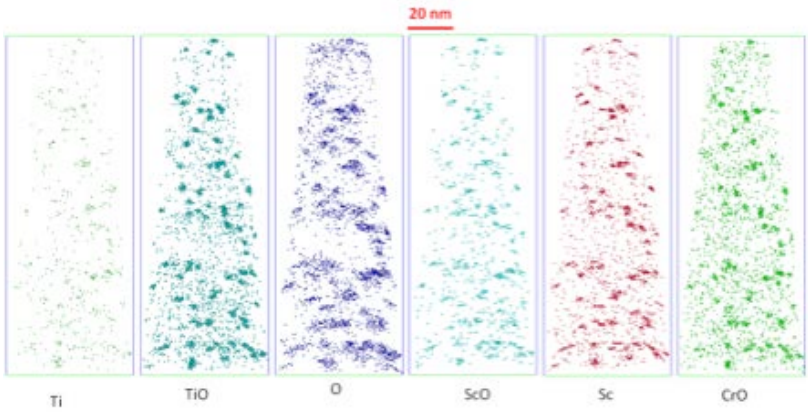

Figure 5. APT maps showing Sc-T-O oxide precipitates in the Sc-based NFA powders aged at 850°C.

Table 1. Precipitate sizes and number densities in the Sc-based versus Y-based (in various conditions) NFAs and using various techniques.

NF Parameters	Technique	US-MA957	Fr-MA957	LTTA	FSW	FSW + Anneal	14SWT 850° C 3 hr	14SWT 1000° C 3 hr
Average diameter (nm)	TEM	3.0	-	-	2.9	3.0	3.3	2.9
	APT	2.3	3.1	2.6	2.2	-	2.1	
	SANS	2.7	2.6	3.9	2.5*	-		
Number density (m^{-3})	TEM	$6.6x10^{22}$	-	-	$1.1x10^{23}$	$1.1x10^{23}$	$8.5x10^{22}$	$7.5x10^{22}$
	APT	$1.8x10^{23}$	$6.0x10^{23}$	$6.7x10^{23}$	$1.6x10^{23}$	-	$2.6x10^{22}$	
	SANS	$9.4x10^{23}$	$7.6x10^{23}$	$2.3x10^{23}$	$3.5x10^{23}$*	-		

Table 2. Average EDS precipitate compositions in the annealed powders.

Anneal Temp (°C)	Avg Sc (at%)	Avg Ti (at%)	Avg O (at%)	Sc/Ti	(Sc+Ti)/O
850	1.68±1.23	2.36±1.47	25.88±4.53	0.72±0.21	0.15±0.07
1000	2.07±0.86	3.17±2.08	30.48±7.37	0.83±0.40	0.17±0.05

Continuing and Future Research

Here we summarize some very preliminary and limited results the potential for replacing Y with Sc in NFA. The next steps will be to: a) SPS consolidate additional batches of milled Sc-based powders at 850°C, 1000°C and 1150°C; b) complete the characterization of all the consolidated NFA conditions; c) determine the reasons for the low hardness in the 1000°C aged powder condition; d) carry out thermal aging studies to assess the stability of Sc-based NFA.

REFERENCES

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