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ANALYSIS OF NANOMETER-SCALE PRECIPITATION IN A RAPIDLY SOLIDIFIED STAINLESS STEEL

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Though stainless steels are important technologically for a wide range of applications, they are not generally known for their very high strength. We have rapid-solidification-processed many stainless steels by gas atomization and achieved strength improvements of over 50% relative to conventionally-processed stainless steels with concomitant improvement in corrosion and oxidation behavior.¹ These strength improvements are most pronounced after aging treatments when elevated concentrations of oxygen and vanadium are present in the stainless steel. An austenitic (FCC) stainless steel (Fe-16%Ni-9%Cr-0.5%Mn-0.2%V-0.0137%N-0.008%O by weight) was prepared by gas atomization and consolidated by hot extrusion at 900°C. These specimens were heat treated for 1 hour at 1000°C and aged at 600°C for 500 hours.

The microstructure of each alloy composition was observed in TEM with bright field imaging. After aging, most alloys showed the same precipitate morphology as before aging. An obvious change, however, was found only in the alloy with highest oxygen content. A high number density ($2.2 \times 10^{20} \text{ m}^{-3}$) of 15 to 20 nm (18 nm average) diameter precipitates was measured in this alloy. Moreover, with weak-beam dark field imaging, a very high number density ($\sim 2 \times 10^{22} \text{ m}^{-3}$) of coherent, 6 to 10 nm (8 nm average) diameter precipitates is observed throughout the matrix by Moiré fringe contrast, Fig. 1a. This image was Fourier filtered to select only the Moiré fringe spatial frequency and the result is shown in Fig. 1b. From the fringe spacing and diffraction pattern, the 8 nm precipitates are FCC with a lattice parameter of 0.40 nm.

These fringes appear only in the aged specimen. An atom probe field ion microscopy (APFIM) investigation showed that FIM provides high contrast imaging the precipitates.² The 18 nm precipitates are complex oxides of Fe, Cr, and Ni with an average composition of 44%O, 31%Fe, 13%Cr, 10%Ni, 3%Ar, Fig. 2. Only one of three oxide precipitates was found to contain argon. Argon was used as the cooling gas in the powder processing. The 18 nm oxide precipitates appear dark with respect to the matrix in the FIM image. The 8 nm precipitates appear bright compared to the matrix and were found to be complex nitrides of Fe, Cr, V, and C with the average composition of 41.5%V, 27.6%N, 18%Cr, 8.6%Fe, 2.4%C, 1%Ni, 0.37%Al, 0.26%Mn.

In order to get a more global view of the structure, energy-filtered composition imaging on a LEO EM 912 was used to map the oxygen and nitrogen in carbon extraction replicas of the aged specimens, Fig. 4. These images confirm that the 18 nm precipitates are oxides, however, it appears that the 8 nm precipitates are not extracted.

References

- 1 J. E. Flinn and T. F. Kelly, US patent application submitted April 1996.
- 2 T. F. Kelly, S. Wisutmethangoon, D. J. Larson, and M. K. Miller, these proceedings.
- 3 This research was sponsored by the Division of Materials Sciences, U. S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp., by the U. S. Department of Energy and was conducted utilizing the Shared Research Equipment User Program facilities at Oak Ridge National Laboratory.

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29

FIG. 1 a) Weak-beam image of Fe-16Ni-9Cr alloy. b) Fourier-filtered version of 1a.

FIG. 2 a) FIM image of a 18 nm oxide precipitate. b) Atom probe profile through the precipitate.

FIG. 3 a) FIM image of a 8 nm nitride precipitate. b) Atom probe profile through the precipitate.

FIG. 4 a) Energy-filtered oxygen image of an extraction replica. b) Vanadium image.

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