

PHASE STABILITY IN CAST HP AUSTENITE AFTER LONG-TERM AGEING

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Casting of high temperature austenitic alloys is often used to form components and structures required in the chemical industry. Alloy HP is a Nb-stabilized austenitic alloy for such applications. High carbon levels are selected in order to drive the formation of coarse, intergranular precipitates of various carbides. These precipitates provide resistance to high temperature creep by inhibiting grain boundary sliding. While these precipitates are present in the cast material prior to high temperature exposure, it is the stability of these second phase particles during ageing that determines the long-term creep resistance and lifetime of stressed components. This study deals with the phase distribution in a centrifugally-cast HP component from a steam superheater tube in a styrene furnace, which experienced temperatures from 927°C to 1066°C or more for over 105,000 h.

The nominal composition of the HP alloy is 35.3 Fe, 32.2 Ni, 28.4 Cr, 1.54 Nb, 1.47 Si, 0.68 Mn, 0.46 C 0.26 N (wt%). Both analytical scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used to characterize the microstructural evolution during ageing. The TEM specimens were prepared either by electropolishing or by mechanical dimpling and subsequent ion milling. The second method produced better thin sections of both coarse precipitates and the adjacent matrix. A backscattered electron SEM image of the aged material shows several precipitate phases (labeled E, M, N) that differ in average atomic number (Fig. 1). X-ray microanalysis in a Philips XL30/FEG SEM was used to identify the major elemental components of each of the phases present (Fig. 2). Low voltage (5 kV) energy dispersive x-ray spectrometry (LV-EDS) permitted the major interstitial impurities to be identified. The darker second phase (M) is a chromium carbide; whereas the intermediate grey second phase (E) contains significant amounts of Si, Nb, Cr, Fe, and Ni with little carbon and no nitrogen. The brightest second phase (N) was rich in niobium; the presence of significant carbon was confirmed by LV-EDS comparison of the 0.1-0.6 keV energy range (where the C K, N K, and Nb M lines overlap) with that of pure Nb. The presence of small oxygen peak in the LV-EDS of all phases was attributed to a thin oxide film on the electropolished SEM samples.

The TEM examination of the aged material provided both quantitative microanalysis (for atomic number $Z > 10$) of the precipitate phases and crystallographic information from selected area electron diffraction. The chromium carbide (M) exhibited relative weight percentages of 100 Cr, 18 Fe, 14 Ni, 1.0 Nb, and 0.2 Si. This composition, as well as that of the other coarse second phase precipitates, may be biased slightly toward the matrix composition as the measurements were performed in self-supporting thin foils of the aged material. Extraction replicas were not employed as a result of the need to confirm the presence or absence of carbon and nitrogen in these precipitates. Diffraction information of the chromium carbide (Fig. 3a) was consistent with face-centered cubic $M_{23}C_6$ with a lattice parameter of ~ 1.05 nm.

The E phase exhibited relative weight percentages of 100 Ni, 68 Cr, 45 Nb, 18 Si, and 11 Fe. The 100 zone of that precipitate phase (Fig. 3b) exhibited systematic absences of reflections with $h+k+l = 2n+2$, indicating a diamond cubic structure with a lattice parameter of ~ 1.10 nm. This composition and the absence of significant carbon indicated that this phase was similar to the silicon-based eta phase observed in other stainless steels and austenitic alloys, rather than a stoichiometric diamond-cubic M_6C phase.^{1,2} However, the silicon-containing eta phase reported contained both molybdenum and niobium. A limited literature search has not located a previous report of an eta phase containing only niobium in austenitic iron-based alloys.

The (Nb, C)-rich N phase exhibited relative weight percentages of 100 Nb, 3.2 Cr, 3.2 Fe, 2.9 Ni, and 0.5 Si. Electron diffraction patterns (Fig. 3c) indicated a face-centered structure with a lattice parameter of ~ 0.43 nm. This lattice parameter is close to pure NbC (0.447 nm). Though there is no niobium nitride with an fcc structure, there is a slightly tetragonal Nb_4N_3 ($a=0.44$ nm, $c=0.43$ nm) that could be present in the aged material. However, this possibility disagrees with the LV-EDS analysis indicated above, where only significant carbon was detected.

Based on the microstructure of related cast alloys, the initial distribution of second phases in the cast HP alloy should be intergranular chromium and niobium carbides. The formation of the eta phase during high temperature exposure indicates that the eta phase is a more stable phase. The high levels of Nb and Si in the HP alloy contribute to eta phase formation. The eta phase links intergranular carbides and engulfs the niobium carbide particles. The continuous layer of intergranular second phases may impact the mechanical properties or corrosion resistance of the alloy.³

References

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3. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Assistant for Energy Efficiency and Renewable Energy, Office of Industrial Technologies (OIT), Advanced Industrial Materials (AIM) Program and the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

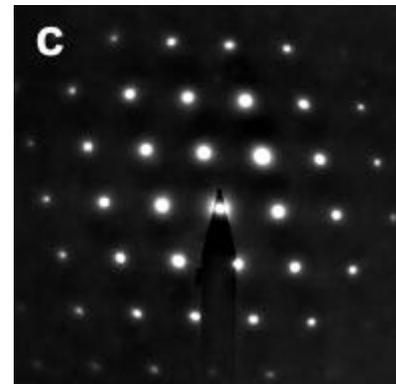
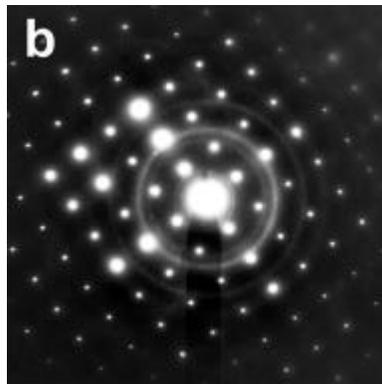
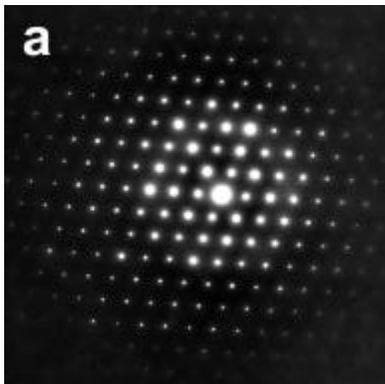
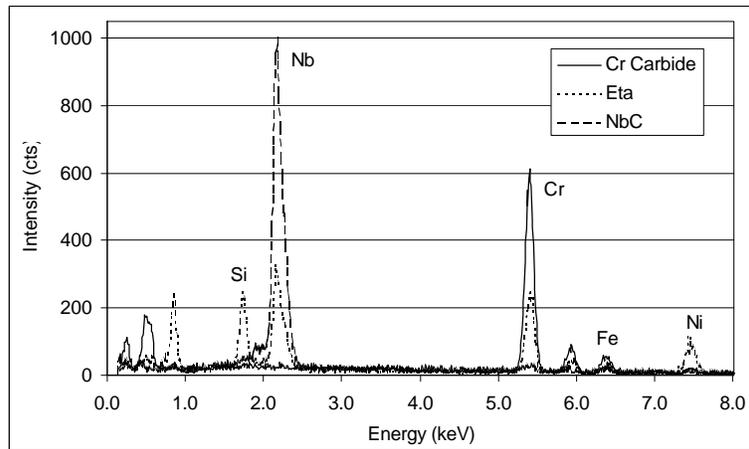
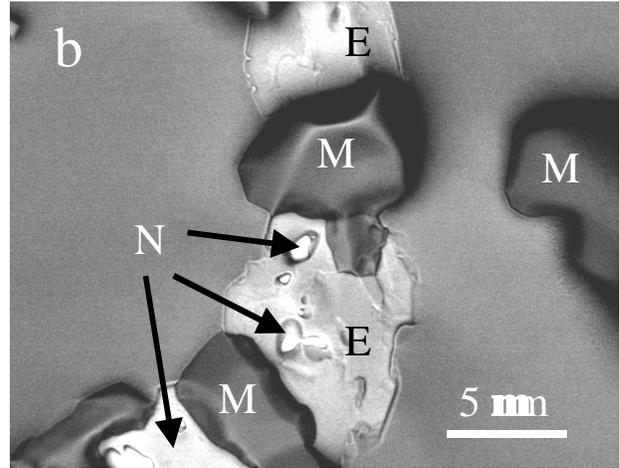
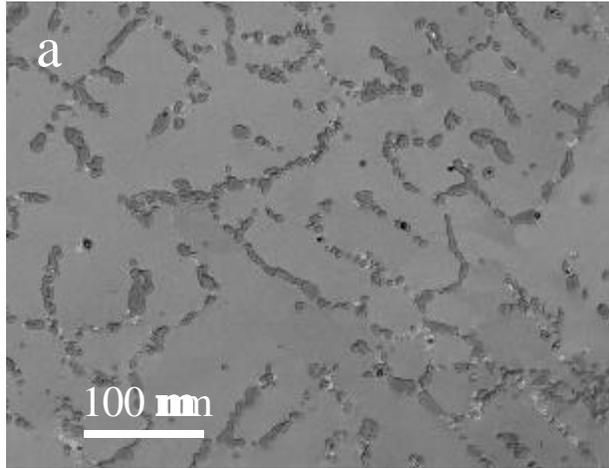


FIG. 1 BSE images of cast HP alloy after 105,000 h ageing. FIG. 2 X-ray spectra of precipitates in aged HP. FIG. 3 SAD patterns from (a) chromium carbide [110], (b) eta [100], and (c) niobium carbide [110] phases.