Effect of Minor Alloying Element Variation on the Properties of Alloy 800

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Application of Alloy 800 in steam generator tubing of fast reactors, where continuous service temperature of the order of 550°C is experienced, has been analyzed with respect to small variations in its chemical composition. Several laboratory melts of Alloy 800 have been prepared and their microstructural and mechanical property changes during simple aging and creep tests at 500 to 600°C have been studied. It has been found that in the above temperature range precipitation of M₂₃C₆ on the grain boundaries is independent of the Ti : C ratio generally specified for Alloy 800. Gamma prime precipitation occurred in alloys containing as low as 0.5 pct Ti + Al after 1000 h of aging and was accompanied with a creep ductility decline. Upon γ precipitation creep rate was retarded and its reacceleration for test times up to 8500 h at 550°C was not observed. Based on the findings, increased Ti concentration at the expense of Al within the specified chemical composition range with carbon content of 0.030 to 0.050 has been suggested.

SUCCESSFUL application of the Alloy 800 in construction of high temperature chemical and fossil fuelled plants, has led to its consideration for sodium cooled fast reactors. The overriding importance of the stress corrosion has guided the nuclear industry to revise the normal Alloy 800 composition. In particular the carbon specification has been reduced from 0.1 pct maximum to 0.03 pct maximum and a minimum value for Ti : C or Ti : C + N is specified to ensure adequate stabilization against sensitization corrosion. More recently stringent control of Ti and Al content has been sought to counter the creep ductility decline observed in long duration tests.

The original ASTM designations of Grades I and II for Alloy 800, now are generally referred to as Alloy 800 and 800 H. The principal difference between the two alloys is the carbon content. Alloy 800 has ≤0.03 pct C and with the final anneal at ~980°C offers optimum mechanical properties for application in the temperature range of 350 to 400°C. Alloy 800 H contains 0.05 pct <C <0.1 pct and is solution treated at ~1150°C to obtain optimum creep properties at above 600°C. For the fast breeder reactors where service temperatures around 550°C are experienced, a compromise between the two specifications, such as 0.03 pct <C ≤0.06 pct, is needed.

The object of the present work has been to study the effect of minor alloying element variation, in particular Ti and Al, within the specified composition range on the properties of the Alloy 800. This work has been conducted with particular reference to the service condition of the Super Phénix reactor (525°C) and future provisions (540 to 550°C).

MATERIAL USED

Six 25 kg laboratory melts of the compositions shown in Table I were received in octagonal ingots which were subsequently:

- hot extruded at 1150°C from 80 mm to about 20 mm diam bars,
- 30 min annealed in argon at 980°C/1000°C,
- cold swaged in three steps to 12 mm diam bars,
- finally heat treated in air for 30 min at 980°C.

To insure quality and homogeneity of the alloys, gamma radiography on the as-received ingots and continuous metallographic observations throughout the fabrication process were conducted. The fabrication steps chosen were with the aim of obtaining alloy grain size of ASTM 8 and the actual grain sizes obtained are shown in Table I.

A commercially produced Alloy 800 and designated here as 3B was also included for parallel tests with the laboratory melts.

EXPERIMENTAL PROCEDURE

Age-Hardening

The bulk of the recent data generated on the Alloy 800 indicate the possibility of structural changes during exposure at high temperatures (400 to 700°C). Therefore, in order to analyze the structural changes occurring in various heats of Alloy 800, simple ageings at 600°C ± 2°C were conducted. For other temperatures, specimens were taken from the screw heads of the creep test pieces, where deformation is not expected.

Creep and Tensile Tests

Constant load creep tests were conducted on high accuracy creep machines (Type TAC, Δl × 100) with a temperature control of ±1°C. Specimens for creep and tensile tests (Ø 4 × 20 mm gage length) were machined from Ø 8 × 40 mm rods utilized for aging.

Metallography

Optical metallography specimens were electrolytically etched in a solution of 7.2 N H₂SO₄ plus a few drops of NH₄SCN (76 g/l of water) with controlled potential at ~150 mV (potentiostat). Thin foils for elec-
electron microscopy were prepared through mechanical polishing of specimens to ~0.06 mm thickness and double jet electrolytic etching (3 or 2.3 mm discs) in a solution of 95 pct acetic acid and 5 pct perchloric acid. The electrolyte bath was refrigerated at –5°C before etch. Surface examination and qualitative analysis of the large precipitates were conducted on the scanning electron microscope equipped with X-ray detector and analyzer.

RESULTS

Macrostructure

The as-received macrostructure of the alloys contained several large intermetallic particles and a series of aligned precipitates. The former were found to be rich in Ti and S and probably have the Ti(C,N,S) composition as reported by previous workers. These precipitates are present in the melt and do not dissolve on subsequent heat treatment at temperatures up to 1250°C. The aligned precipitates were identified by electron diffraction patterns to be M_{23}C_6 phase (primary M_{23}C_6) in general. During early stages of aging new M_{23}C_6 precipitates were formed (secondary M_{23}C_6) principally on the grain boundaries, and grew in size with prolonged aging time. A typical photomicrograph showing M_{23}C_6 grain boundary precipitation in Alloy 800 is presented in Fig. 1. No marked difference in the microstructure of the alloys was noted for aging times up to 3000 h.

Precipitation Hardening

Alloy response to aging as measured by hardness and tensile tests is presented in Fig. 2 and Figs. 3(a) and (b) respectively. Although the aging time in this work does not exceed 10^{4} h, our in-house data on other heats of Alloy 800 indicate a plateau in the age-hardening curve at ~10^{4} h of aging at 550 to 600°C. The precipitation strengthening was found to be a function of Ti or Al (with Al or Ti constant respectively) or Ti + Al concentration, as shown in Fig. 4. The curve obtained was in good agreement with the reported data by other investigators, as indicated by letters S, E, X and M on the Fig. 4. Nevertheless, it should be pointed out that although the general trend of the addition of each element was towards higher hardness, markedly scattered results were obtained during rapid age-hardening period with higher Ti + Al concentrations (Alloys 3, 5, 6 and 3BI). In such cases both points are shown in Figs. 3(a) and (b).

Microhardness tests conducted inside the grains of the alloys, associated the hardening with the matrix. Electron microscopy confirmed precipitation hardening through γ′(Ni_{3}Ti,Al) in all alloys except 4. Extensive γ′ precipitation was detected in the Alloys 3, 5, 6 and 3BI by 1000 h of aging at 600°C (Fig. 5), whereas longer times were needed for the Alloys 1 and 2 (Fig. 6). The γ′ precipitates in the higher Ti + Al concent-

![Fig. 1—Typical photomicrograph of Alloy 800 (Ref. 3) after 3000 h at 600°C.](image)

![Fig. 2—Hardness (Vickers) vs aging time at 600°C.](image)