## **Electron Microscopy Of Heat-Resistant Alloy Sheets And Foils Being Considered For Use In Recuperators**

N.D. Evans, P.J. Maziasz, Y. Yamamoto, and J.P. Shingledecker

Metals & Ceramics Div., Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831

Microturbines are an attractive alternative for both distributed power generation and combined heat and power applications. Recuperators are compact, high efficiency heat-exchangers that improve the efficiency of smaller gas turbines and microturbines. While traditionally made from 347 stainless steel and operated below or close to 650°C, recuperators today are being designed for reliable operation above 700°C, where failure mechanisms may include creep due to the foil's inherent fine grain size, accelerated oxidation due to moisture in hot exhaust gasses, and loss of ductility with aging [1]. Research on both commercially available and developmental alloys has been conducted to identify cost-effective, capable recuperator materials for the next generation of advanced microturbines. High-temperature alloys examined in this study include: commercially available foils (typically 100 μm thick) and sheets (typically 250-375 μm thick) of 347 steel, HR120, lab-scale processed foils of NF709 and 625, and AL20-25+Nb. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) studies were performed on both asreceived specimens and gage-portions of creep-ruptured specimens. Phase identification was accomplished by combined diffraction studies and energy dispersive X-ray spectrometry (EDS).

Figure 1 contains creep-rupture data from tests conducted at  $750^{\circ}\text{C}$  and 100 MPa. Creep-rupture of 347 steel, which failed after only 50 h, is attributed to rapid formation of the brittle Fe-Cr  $\sigma$  phase at grain boundaries (Fig. 2: 347 steel crept at  $704^{\circ}\text{C}$  and 150 MPa. Here, the behavior of the 347 was similar at both creep conditions relative to the other alloys). In comparison, creep-tested alloy 625 developed a uniform dispersion of Si-Mo-Cr-Ni M<sub>6</sub>C precipitates along grain boundaries, and a dense lath structure within each grain which contribute to its creep-strength (Fig. 3). The NF709 (Fig. 4) developed a uniform dispersion of Si-Mo-Cr-Ni M<sub>6</sub>C and M<sub>23</sub>C<sub>6</sub> precipitates along grain boundaries, whereas nearly all precipitates developed along grain boundaries in HR120 during creep (not shown) are M<sub>23</sub>C<sub>6</sub>, with few M<sub>6</sub>C. Fine NbC precipitates formed in grain interiors of both NF709 and HR120 during creep-testing (Fig. 5). The alloy AL20-25+Nb (Fe-20Cr-25Ni + Mo, Nb, N), similar to NF709, is being developed for commercialization and has been examined in the uncrept condition. Here, in addition to the expected fine NbC precipitates (Fig. 6a) observed in the as-processed condition, AlN precipitates were formed (Fig. 6b), as identified by both diffraction patterns (e.g., B= [0001] in Fig. 6c) [2] and EDS (Fig. 6d). Results of creep-tests and the microstructure developed during creep of this alloy will also be discussed.

The improved creep-resistance of alloys 625, HR120, and NF709 relative to the standard 347 steel at 650-750°C can be attributed to the formation of grain boundary phases which increase creep strength rather than embrittle the alloy. Additionally, HR120 and NF709 form fine NbC precipitates which contribute to dispersion strengthening, and similarly, alloy 625 develops a dense, stable lath structure within each grain. These alloys should provide enhanced performance and temperature capability of recuperators at about 2-4 times the cost of 347 steel [3].

## References

- [1] P.J. Maziasz et al., ASME paper GT-2005-68927 Am. Soc. Mech. Engin. New York, NY (2005)
- [2] C. Yeh et al., Phys. Rev. B 46, (1992) 10086–10097.
- [3] Research supported (NDE, PJM, YY, JPS) by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Distributed Energy and Electrical Reliability, and (ORNL SHaRE User Facility) by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

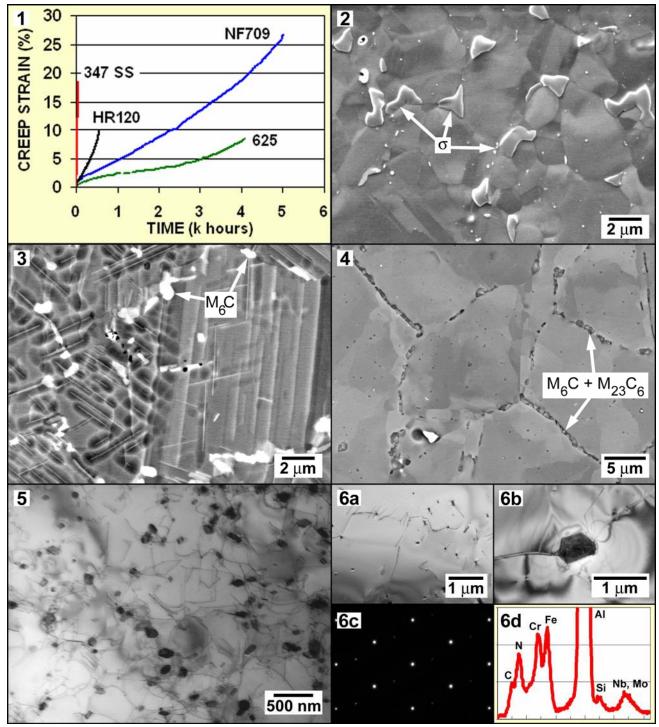


Fig. 1. Creep-rupture test results of alloys being considered for next generation recuperators.

- Fig. 2. Sigma phase formation at grain boundaries of crept 347 stainless steel.
- Fig. 3. Crept alloy 625 has a stable dispersion of M<sub>6</sub>C precipitates along grain boundaries.
- Fig. 4. Crept alloy NF709 developed M<sub>6</sub>C and M<sub>23</sub>C<sub>6</sub> precipitates along grain boundaries.
- Fig. 5. Fine NbC precipitates in NF709 developed during creep testing.
- Fig. 6. Uncrept AL20-25+Nb a.) fine NbC precipitates b.) AlN precipitate
- c.) SAD pattern, B = [0001], and d.) N K and Al K EDS peaks from AlN in Fig. 6b.