I appreciate your sending me a copy of your thoughts on cracking. I have a few comments, mostly supplementary in nature. The numbers by each comment refer to certain locations in text.

(1) Probably difficult to say that any component removed from the MSRE was unstrained.

(2) Tests on sampler cage rod shows cracks to depth of at least 3 grains. Otherwise your statement is true.

(3) Really first obvious in specimens removed in June 1969 and described in TM 3063. Many words in this report about cracks, but items summarized on pp. 79-82 are most relevant (particularly Item 8 on p. 82).

(4) I would assume here that you are thinking of loops, etc. From what I can learn, a natural circulation or pump loop was never run in which the oxidation state of the salt was known. Loops such as DeVan's when FeO was added, should have been more oxidizing than others. However, no numbers are available for such parameters as the $\frac{U^{3+}}{U^{4+}}$ ratio.

(5) Another important difference between the MSRE and in-pile experimental loops is that no in-pile loop ever ran for much time with fission products available. Compere's and Trauger's loops generally lasted 1000 hr or less without failure. The first group of surveillance specimens from the MSRE had been at temperature 4800 hr with some concentration of fissions products all the time. Cracks were only beginning to form. Thus the loops may not have had sufficient exposure. None of the loop and capsule parts were deformed as test except Compere's. We were able to produce cracks in the cooler (545°C) section.

(6) Metallurgical Transactions, vol 2, 1492 (1971). Note that there was a clear source of strain here because of the phase transformation in iron on cooling.

(7) Not sure that "corrosion" is the correct word here. Tellurium caused the grain boundaries to be brittle so that they cracked when the specimen was strained. No evidence of "corrosion" until strained.
Many of the corrosion experiments to date to which an oxidant has been added have resulted in selective grain boundary attack. This makes the grain boundaries visible in the as-polished condition, but they do not crack when the specimen is strained. Thus, we have produced a grain boundary modification, but not the cracking.

There are several variables that need to be understood before tying down the conditions for an in-reactor experiment. The following come to mind.

a) Continue $^{3+}/^{4+}$ controlled loop to see whether selective corrosion can be produced that leads to grain boundary embrittlement.

b) Stressed experiments (tube burst) should be run to determine whether Te has the same effects when salt is reducing and oxidizing.

c) Diffusion measurements should be made to determine how rapidly Te diffuses along the grain boundaries of Hastelloy N. This will help set the time required for experiments.

d) Schaffer's work is being expanded to include Ni, 304 stainless steel, and Hastelloy N. This will give an indication of the effects of composition on the cracking caused by exposure to Te.

HEM:kg

Attachment

cc: M. W. Rosenthal
J. R. Weir
Effects of Fuel Salt on Hastelloy N

What

What are we worried about? What have we seen?

A kind of intergranular attack that destroys the cohesion between grains near the surface of Hastelloy N, sometimes creating perceptible indications (1) in unetched, unstrained specimens, certainly making the boundaries susceptible to separation upon tensile strain. It extends at most about one grain deep in all cases observed so far. (McCoy and McNabb are preparing a memo or two to answer these questions in detail.)

Where

Where did this problem crop up?

It first came forcibly to our attention when we inspected portions of (3) Hastelloy N equipment that had been exposed to the fuel salt throughout the MSRE operation. Hastelloy N specimens removed earlier from the fuel system had also shown it (to lesser degrees).

Was it universal in the fuel system? Or was it confined to particular heats of metal or certain situations (flow, temperature, fission density)?

Universal, as far as we have been able to tell. It appears on tubes in the heat exchanger as well as on rod thimbles and specimens in the core. Specimens of various heats exposed during different intervals of MSRE history showed the same phenomenon. (The degree of attack varied, but the same mechanism appeared to have been operating.) Different sets of specimens were exposed for different durations (whether measured in hours or integrated power). Some were exposed only during intervals when oxidizing potential was off one way or the other from the lifetime average.
Where else in the MSRP had the same phenomenon been evident?

Nowhere else, as far as we have been able to determine, had there been a situation that we can say positively is the same phenomenon. There were some cases of attack that bear resemblance in some way or another, but none with all the characteristics.

The situations in most respects closest to the MSRE fuel system conditions did not show the same kind of attack. The other sides of the heat exchanger tubes and the control rod thimbles were the same piece of metal, at virtually the same temperature as the fuel side, and didn't crack, putting the finger on the fuel salt. Some of the specimens in the control test stand were of the same heats of Hastelloy N as some of the fuel system pieces, were exposed to salt of the same (nominal) chemical composition, saw close to the same temperature-time history but didn't crack.

Inferred Causes

The reason(s) for cracking or not must lie in the few differences between those situations similar to the MSRE but without cracking and the situation in the MSRE fuel system. What were those differences?

The MSRE fuel salt contained fission-product atoms in some form. (Highly ionized at birth, they pick up electrons from their surroundings to get to final valence state.)

Nothing else? How about oxidation-reduction potential?

There were greater differences between the potential in the fuel system during exposures of different sets of core specimens which all showed cracking than there was between the fuel system when the attack was occurring and other systems where this kind of attack didn't occur. More to the point, attack occurred in the fuel system when the reducing conditions were the same as in other systems where attack did not occur (I think). (This is important; find out for sure.)
How about processes related to high-velocity particles and atoms emitted and recoiling in the fuel salt?

The fluxes of these at surfaces in the core were much higher than at the surfaces of heat exchanger tubes which cracked as badly. Also, it seems unlikely that this kind of process is consistent with the selective attack at the grain boundaries.

Other Pertinent Experience

Hasn't anyone ever seen this same thing elsewhere?

Probably. Certainly some cases have been observed that bear suspicious likenesses both in the appearance of the effect and in the existence of the same possible causative factors.

An adverse effect of tellurium on the intergranular cohesion in iron was observed at the North American Rockwell Science Center. An alloy made up with 0.02 wt % Te cracked on quenching and Auger spectroscopy showed that tellurium represented about 25 at % of the fracture surface composition. (An article on this work was submitted to AIME in 1970.) The cracks here were throughout the specimen, not just near the surface as in the MSRE, but could represent pervasive existence of the same cause that existed only near the surface of the MSRE Hastelloy N. The NARSC investigators refer to known similar intergranular embrittlement by other elements.

A 1963 article in Metallurgia by investigators from the International Nickel Co. reports that incorporation of traces of various elements highly damage the rupture properties of nickel-base alloys at high temperature. The nature of the failure was not explained but the authors conjecture that the harmful elements must have concentrated in or near the grain boundaries. Fifteen elements were added; four had notable effects; tellurium was the worst.
The current ORNL experiment in which various elements were vapor-deposited on Hastelloy N has already shown that tellurium produces visible evidence of corrosion on specimens held for 1000 hr at 650°C in evacuated ampoules.

Some specimens corroded in fluoride salt in an oxidizing state show effects that more or less resemble the intergranular effect observed in the MSRE fuel system.

**Investigations**

What is being done to investigate (and cure) the problem?

Various out-of-pile experiments are either underway or are being planned within the MSRP by M&C and Reactor Chemistry personnel. In-pile experiments have also been suggested, and a tentative decision has been made to proceed with them.

**In-Pile Experiments**

What do we hope to accomplish?

To prove the cause and the cure. Other experiments may point the way, but we need this proof to justify continuing the MSRP.

How?

Expose capsules containing Hastelloy N specimens in fuel salt in which the reducing potential and impurity levels are in desired ranges and accurately known. We don't know yet exactly what we will want to try. All we can say is that we may change (slightly) the salt chemistry and try specimens of various alloys.

Can we specify conditions now?

Only generally. Salt compositions like in MSRE and in MSBR. Temperatures between 550 and 750°C. Pressures low. Fission rate to give, within a reasonable period (1 to 3 ORR cycles?), fission product inventories per unit surface area like those in MSRE.