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SUBJECT: Operation of the HRT Mockup with Boiling Fuel
in a Titanium Pressurizer, Run CS-23
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SUMMARY

The 0.045 M UO_2SO_4 , 0.036 M CuSO_4 , 0.025 M H_2SO_4 solution (HRT fuel composition) was chemically stable during 1,866 hr of operation at 280°C and 1500 psi. The system was pressurized by boiling a 0.4 gpm stream of the fuel in a titanium heat exchanger at 313°C.

During 1,656 hr of operation with the loop at 300°C, three excursions were made to pressurizer temperatures above 330°C where two liquid phases were formed. These tests indicated that heavy phase began formation at 325°C (vapor pressure equilibrium temperature) as evidenced by loss of fuel from the circulating stream. Good heat transfer excluded the possibility of the missing material depositing in the form of a scale in the heat exchanger. In each test, the original fuel composition was restored within six hr after the pressurizer temperature was lowered below 325°C.

The generalized stainless steel corrosion rate during operation at 280°C and 1500 psi was 0.6 mpy for the first 700 hr and 1.6 mpy for the next 1166 hr. The average rate during the period when excursions were made into the two-phase region was 3.0 mpy. The apparent increase in corrosion rate is not easily explained because no unusual attack could be found on inspection of the stainless steel surfaces, particularly in the vicinity of the titanium heat exchanger.

Boiling fuel solution in a titanium heat exchanger appears to be a practical source of steam for pressurizing an aqueous homogeneous reactor.

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OPERATION OF THE HRT MOCKUP WITH BOILING FUEL IN

A TITANIUM PRESSURIZER, RUN CS-23

The objective of this test was to investigate the feasibility of steam-pressurizing an aqueous uranyl sulfate fuel system by boiling the fuel in a titanium heat exchanger. The low corrosion rate of titanium found under these conditions,¹ compared to the high corrosion rate and fuel instability encountered with boiling fuel in stainless steel,² renewed interest in this method of pressurization.

The HRT Mockup Facility

The mockup facility comprises a high pressure circulating system connected to a low pressure system through a let-down valve (and titanium heat exchanger) and a feed return through a diaphragm type pump.^{2,3} The high pressure system was modified by the addition of a titanium heat exchanger and vapor separator connected so as to boil a small side stream of fuel from the circulating loop, discharging the steam to the original stainless steel pressurizer and returning liquid to the loop, as shown in Fig. 1. The exchanger consisted of 30 ft of jacketed, 1/2-in. A-40 titanium pipe, and the vapor separator was 8 ft of 3.875 in. OD, 0.355-in. wall, titanium A-110-T pipe capped at each end. The heat exchanger jacket received Dowtherm vapor from a boiler at a temperature about 7 to 10°C above the pressurizer temperature.

Special transition flanges⁴ were used to connect the titanium pipes to the stainless steel loop, and the addition described brought three more of these connections to the facility, raising the total number to nine. An orifice at the inlet transition flange to the heat exchanger restricted the flow to the pressurizer.

Operation

The test was made with a fuel composition of 0.045 m UO_2SO_4 , 0.036 m CuSO_4 and 0.025 m H_2SO_4 , which is the nominal composition of the HRT fuel. The test was divided into two parts, the first of which consisted of operating for 1866 hr at a circulating fuel temperature of 280°C and a pressurizer temperature of 313°C (1500 psi). Since samples of fuel heated in quartz tubes formed a second liquid phase in the range of 328° to 332°C, the second part of the test, of 1656 hr duration, involved three excursions to pressurizer temperatures of 330°C and above. The circulating loop was held at 300°C during the excursions. In two of the excursions, at a pressurizer purge flow of 0.4 gpm, the temperature was raised successively to 325°, 330° and 336°C, obtaining equilibrium data over periods from one to four days at each temperature. The third excursion was made in five-degree increments from 320° to 330°C with a pressurizer purge flow of 0.2 gpm.

Results

During the 1,866-hr run at 280°C and 1500 psi, the sample analyses were consistent. There was no material lost from solution, as was noted in run 22.⁵ Boiling the fuel solution at 313°C in the titanium heat exchanger did not affect the fuel stability.

During the three excursions to higher pressurizer temperatures it became apparent that the second liquid phase began formation at a vapor pressure equilibrium temperature of 325°C in the pressurizer. This is not surprising because the heat exchanger wall temperature was estimated to be several degrees higher than this. Each time that the temperature was increased the amount of heavy liquid phase formed in the heat exchanger was inferred from the reduction in uranium concentration in the light phase circulating through the rest of the system. The phenomenon was reversible with temperature inasmuch as the original fuel composition was restored within 6 hr after the temperature was lowered below 325°C. Good heat transfer excluded the possibility of the missing material depositing in the form of a scale in the heat exchanger. The analytical results of samples withdrawn from the circulating stream are shown in Fig. 2. The analyses are consistent in the second and third excursions, but the free acid increased unaccountably in the first excursion.

Typical equilibrium analyses of the circulating stream for operation at the various pressurizer temperatures were calculated from Fig. 2 (second excursion) and are shown in Table 1 along with the calculated volumes of the heavy phase that formed, assuming a concentration of 800 g of U per liter in the heavy phase.

Table 1. Circulating-Fuel Composition in HRT Mockup at Several Pressurizer Temperatures

Pressurizer Temp (°C)	Molal Composition				Heavy-Phase Volume	
	U	Cu	SO ₄	2H ⁺	Liters	% of Heat Exchanger Tube Volume*
320	0.0445	0.0358	0.1053	0.025	0	0
325	0.0422	0.0348	0.1030	0.0265	0.09	6.4
330	0.0348	0.0305	0.0930	0.0265	0.38	27.0
336	0.0290	0.0270	0.0832	0.0270	0.66	47.0

*It is believed that the heavy phase was confined to the heat exchanger because the temperature there was high enough for its existence and there was no local attack of the adjacent stainless steel.

Because the fuel velocity through the heat exchanger might have affected the fuel stability, the flow was lowered to 0.2 gpm in the third excursion by changing the orifice in the heat exchanger inlet. During the startup the solution was inadvertently boiled in the heat exchanger without liquid flow to the pressurizer, resulting in the formation of a partial plug due to precipitation of salts. The plug was not removed by resumption of some flow under steam pressurization, and finally it had to be removed by dropping the pressurizer temperature and operating under gas pressurization at 200°C (i.e., no evaporation in the heat exchanger). The fuel composition and heat transfer returned to normal, and the system continued operation in routine manner.

Lowering the flow to the pressurizer had no apparent effect on the fuel stability, except possibly on the nickel behavior described below.

The generalized corrosion rate for the stainless steel system, inferred from the rate of increase of nickel in solution, was $0.6 \pm 0.1^*$ mpy for the first 700 hr of operation and then increased to 1.62 ± 0.15 mpy during the next 1166 hr of operation. No reason for the change in rate was found. During operation with the pressurizer temperature above 325°C the nickel analyses became so scattered that no direct conclusion could be drawn regarding the corrosion rate. However, on returning to lower temperatures, the increase in nickel with time became apparent again, and an average rate for approximately 91 days of operation (including the three excursions) was statistically determined after discarding analyses of samples taken during operation above 325°C or during known system upsets. The rate so determined was 3.02 ± 0.17 mpy, or nearly double the previous high rate. It is hard to believe that the indicated increase was due to systematic error, but subsequent inspection of the high pressure system, particularly those stainless steel parts in proximity to the titanium heat exchanger, failed to reveal evidence of unusual corrosion.**

If interest continues in this method of pressurization for the HRT, the present run should be repeated, probably with fresh fuel, to determine if the increased corrosion rate is real and if it is related to operation above the two-phase formation temperature of the fuel.

*Limits of error at the 0.05 level of certainty.

**About 20 g of solids were removed from the entrance to the titanium heat exchanger at the end of the run. The solids were mainly chrome and iron oxide with about 5% titanium.

Miscellaneous Equipment Performance

The let-down valve operated well during the entire run. This valve had a 1/8-in. port and a maximum C_v of 0.064. At a 1 gpm flow the valve operated 70% open with a C_v of 0.025. The valve was leak tight with 2000 psi across the seat after the run. The pressurizer level controlled the valve position except for about 1000 hr when the valve was set manually and the pressurizer level controlled the feed rate.

An all-stainless steel Brooks variable area flow meter was used to measure the circulating pump purge flow. This rotameter, which is being evaluated for reactor use, operated for 2000 hr without maintenance. The pulsating action of the purge pump was dampened by a throttling valve and constant level head tank.

The operation of the feed pump was good. For 1000 hr of the run the feed rate was controlled by the pressurizer level with the let-down valve set manually. A diaphragm failure occurred near the end of the run. The diaphragm was 0.021 in. thick, 1/4 hard, 347 stainless steel which had been in service for 12,875 hr. The diaphragm was protected by a 40 mesh wire cloth filter. The pump head has been replaced but the head that failed has not been cut open for examination yet. The new head is of a later design and is expected to have a longer life.

The nine stainless steel-to-titanium transition flanges remained leak tight during the 3,522-hr run. Two pairs of these flanges were disconnected and reconnected five times during the run.

The Pressure Products oxygen compressor operated intermittently for 700 hr during the run and supplied oxygen to a 2200 psi header for continuous metering into the mockup. The intermittent operation in earlier tests allowed the leather packing around the piston drive to dry out between operating periods. This led to leaking and gas binding of the compressor. Before this run the piston drive was replaced by a Scott and Williams oil drive which was separated from the all welded water intermediate system by a rubber pulsator in a flanged housing. Operation was satisfactory.

Conclusions

So far as can be determined from the tests reported here, boiling fuel solution in a titanium heat exchanger appears to be a practical method of obtaining steam for pressurization of an aqueous homogeneous reactor.

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Fig. 1 HPL Mockup Showing Titanium Pressurizer

Fig. 2 Fuel Concentration vs Time at Various Pressurizer Temperatures

Loop Temp 300°F

O - Uranium

Δ - Copper

X - Free Acid

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