

TESTING OF ULTRAFILTRATION MEMBRANES FOR APPLICATION IN A GENERATION IV- SUPER CRITICAL WATER REACTOR

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Abstract

The conceptual Supercritical Water Reactor (SCWR) uses supercritical water as the working fluid. SCWRs are designed for operating at high pressures and temperatures in order to achieve a higher thermal efficiency of about 45% vs. about 33% for Light Water Reactors (LWRs). It is expected that methods for purifying the corrosion products from SCW are required, including ultrafiltration of corrosion products particulates. We have now undertaken some experiments to test the stability of several filter membranes at high pressures and temperatures for application in the potential GEN-IV plant. A SCW flow set-up has been built. Initial microscopic analysis of the membranes has been performed and the results of this work are presented here.

1. Introduction

The Generation-4 program is a current international research program with the aim of creating and constructing new nuclear power plant (NPP) designs. The primary goals are to improve nuclear safety and to protect the environment. It is expected that the new reactors will be online by 2030. Six different reactor systems have been selected for evaluation:

- Gas-Cooled Fast Reactor (GFR)
- Lead-Cooled Fast Reactor (LFR)
- Sodium-Cooled Fast Reactor (SFR)
- Molten Salt Reactor (MSR)
- Very-High-Temperature Reactor (VHTR)
- Supercritical-Water Reactor (SCWR)

The AECL, McGill University and the University of Saskatchewan are working together to develop and realize a SCWR project. The supercritical water reactors are using supercritical water as the working fluid. They are designed to function at higher pressures and temperatures whereby a higher thermal efficiency (about 45% vs. about 33% LWRs) can be achieved [1].

A problem has occurred for fossil-fired boiler tubes operating under SCW conditions. Heavy corrosion product deposits are produced in the tubes [2]. For this reason, methods for purifying the corrosion products from SCW are required. We are researching different kinds of filter materials. The focus of our research project is to find a membrane which is stable enough and

able to clean the water in the SCWR. Nanofiltration or ultrafiltration can be used to remove a single component or a group of components from a liquid mixture [6]. To realize our project, we have built a setup that is working under supercritical water conditions.

2. Experimental

The setup is made up of a pump, a two-way shut-off valve, a relief valve, a furnace, a pressure reducer, a check valve, a heat exchanger, two pressure gauges and four thermocouples.

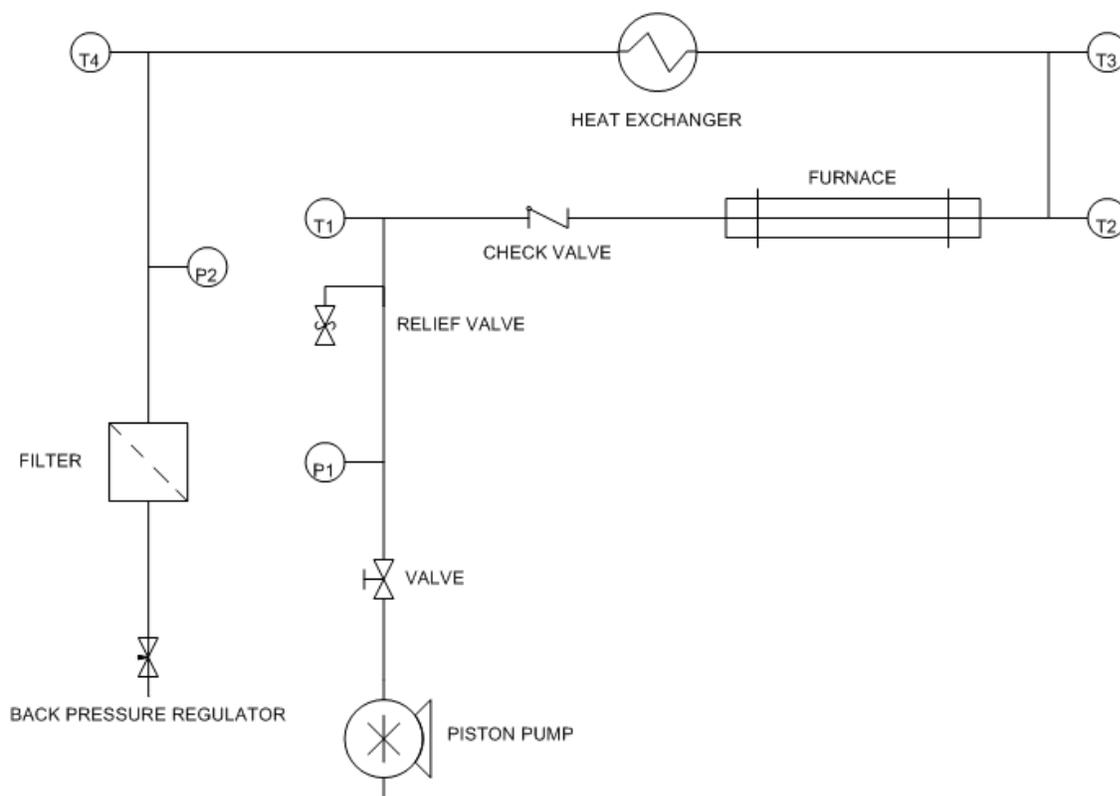


Figure 1

2.1 Pump

In order to obtain a constant flow rate, a HPLC (High performance liquid chromatography) pump is used. The flow rate of the pump goes from 10 $\mu\text{L}/\text{min}$ to 200 mL/min.

2.2 Two way shut off valve

The 1/16" two-way shut-off valve is designed so that the flow can be shut off if there is an abnormal operation or an accident. It is designed for high pressure, low volume systems.

2.2 Relief valve

The relief valve is a proportional relief valve that opens if the pressure increases to about 4000 psi.

2.3 Furnace

The furnace is designed for a temperature of 600 °C.

2.4 Pressure reducer

The pressure reducer used to obtain pressures up to 250 bar is a spring-loaded back-pressure regulator. It opens when the system pressure exceeds the set point and bleeds off working fluid at the rate required to maintain the system pressure at the set point.

2.5 Check valve

The check valve limit reverses the flow.

2.6 Heat exchanger

The heat exchanger cools down the water.

2.7 Pressure gauge

The pressure gauges monitor the pressure in the tubes before the furnace and after the heat exchanger.

2.8 Thermocouples

The thermocouples monitor the temperature in the water system.

2.9 Furnace Control system

The furnace control system monitors the temperature in the furnace. The system is set up so that the furnace reaches and stays the pre-set temperature of 600 °C.

2.10 Shielding

The shielding material is 3/8" polycarbonate, which provides a resistance to projectiles with an energy < 5.6 ft.lbs.

2.11 Tubes

We are using 316 stainless steel tubes.

2.12 Fumehood

The laboratory fume hood assured a clean environment. The fume hood sashes act as an additional protecting shield.

2.13 Safe Operating Procedure

For the operation of the supercritical water setup, we developed a Safe Operating Procedure (SOP)

Start-up / pressurization

1. Switch on power to pump
2. Open the 1/16" valve, and prime the pump with a flow of water to remove air bubbles
3. Set the required flow rate on the pump
4. Set the required operating pressure with the back pressure regulator
5. Initiate data collection system (turn on computer and setup the software)
6. Test system for leaks
7. TURN ON COOLING WATER

Heating

1. Switch on the furnace
2. Set furnace trip temperature to appropriate level, e.g., 20 °C > operating T
3. Increase the heater setpoint step-by-step up to operating temperature

Standard shutdown

1. Set heater set-points to 0 °C
2. Once heater and fluid temperatures < 80 °C, switch off pump and furnace
3. TURN OFF COOLING WATER
4. Turn off data collection system

Emergency shutdown procedure: Turn off pump and furnace, leave cooling water on.

(If possible, it is preferable to have the heater setpoint set to 0 °C to allow the furnace temperature to be monitored after shutdown.)

THIS EQUIPMENT MAY ONLY BE USED BY APPROPRIATELY TRAINED OPERATORS.

2.14 Safety Standards

The setup meets Canadian safety regulations and standards. Safety precautions are the shielding, the SOP, the relief valve, the furnace control system (set point control high limit) and the cooling cycle. The furnace is labeled for Canadian electrical specifications. The fume hood guaranteed a clean environment.

3. Results and Discussion

We have analyzed different stainless-steel membranes with porosities of 0.5-1.0 μm from different companies. To analyze them, we put the filters in the $\frac{1}{4}$ inch tube between a nut and a fitting inside of the furnace and five analysis methods were used: the Nanoanalyzer-, the Backscatter-, the SEM-, the Profilometry- and the EDS-method.

3.1. Nanoanalyzer

NanoAnalyzer is a scanning force microscope that works in a regime of rigid contact and does not require a vacuum. The main characteristic feature of NanoAnalyzer is the use of piezoresonance probe with high bending stiffness of the cantilever. Use of the regime of resonance oscillations permits checking of contact between the probe tip and the surface on two parameters to be performed: change of amplitude A and frequency F of the probe oscillations [5].

3.2 Backscatter

Backscattered Electron Detectors are typically placed above the sample in the sample chamber based on the scattering geometry relative to the incident beam. The detectors are solid-state devices, often with separate components for simultaneous collection of back-scattered electrons in different directions. Detectors above the sample collect electrons scattered as a function of sample composition and detectors placed to the side collect electrons scattered as a function of surface topography [7].

3.3 Scanning Electron Microscopy (SEM)

With the SEM-method the morphology of membrane surfaces and cross sections can be analyzed [3].

3.4 Profilometer

The profilometer has proved to be a highly versatile and accurate instrument for the measurement of true 3D objects of almost any material. The measuring principle, based on the compact disc laser sensor, is noncontacting and thus suitable for sensitive and fragile surfaces, except where surface roughness is excessive. Most importantly, the rotating sensor head, together with the inclination feedback signal from the sensor, overcomes the disadvantage of conventional types of profilometers, which can only scan across a relatively flat surface [4].

3.5 EDS spectra

Electron probe X-ray microanalysis technique – energy-dispersive X-ray spectroscopy (EDS) – uses the characteristic X-rays generated from a sample bombarded with electrons to identify the elemental constituents comprising the sample. The technique generates a spectrum in which the peaks correspond to specific X-ray lines and the elements can be identified [8].

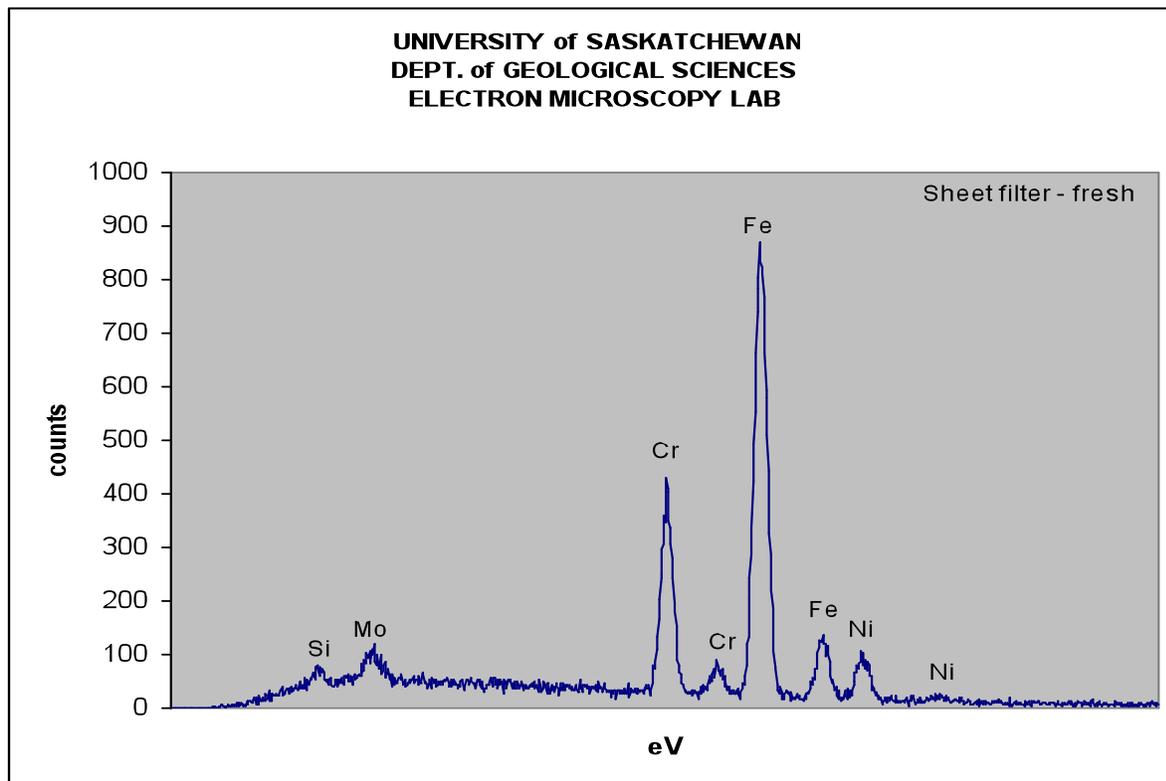
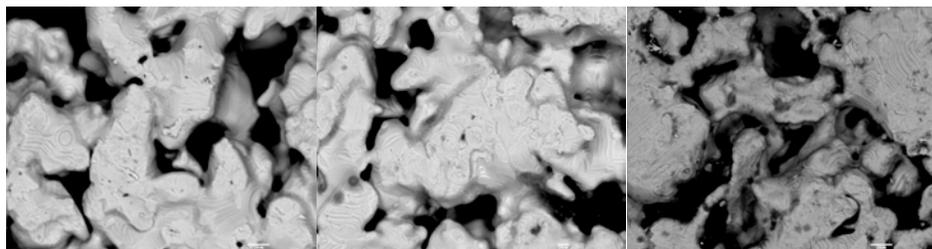


Figure 2

The material of the membranes are composed of Si, Mo, Cr, Fe, Ti, C and Ni. It seems that this composition of elements is suitable for designing filters that should be resistant to supercritical water.

The quality of the methods is different. The best results regarding the texture were obtained using the backscatter- and the SEM-methods. Below are illustrated three pictures for both methods, one of the original membrane (unused) with the pore size of 0.5 μm , one of the membrane under subcritical conditions and one under supercritical conditions (250 bar, 390 $^{\circ}\text{C}$). The changes in structure and texture are not significant.

Backscatter-technique



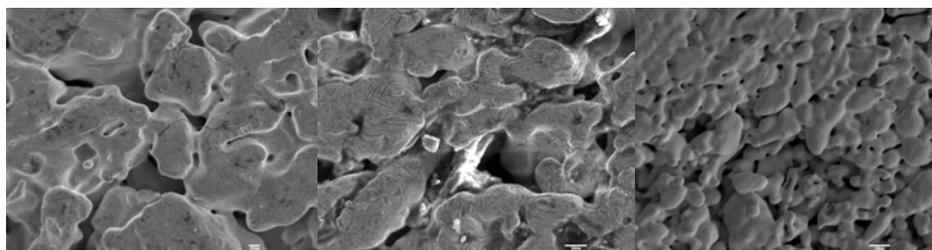
unused

subcritical

supercritical

Figure 3

SEM-technique



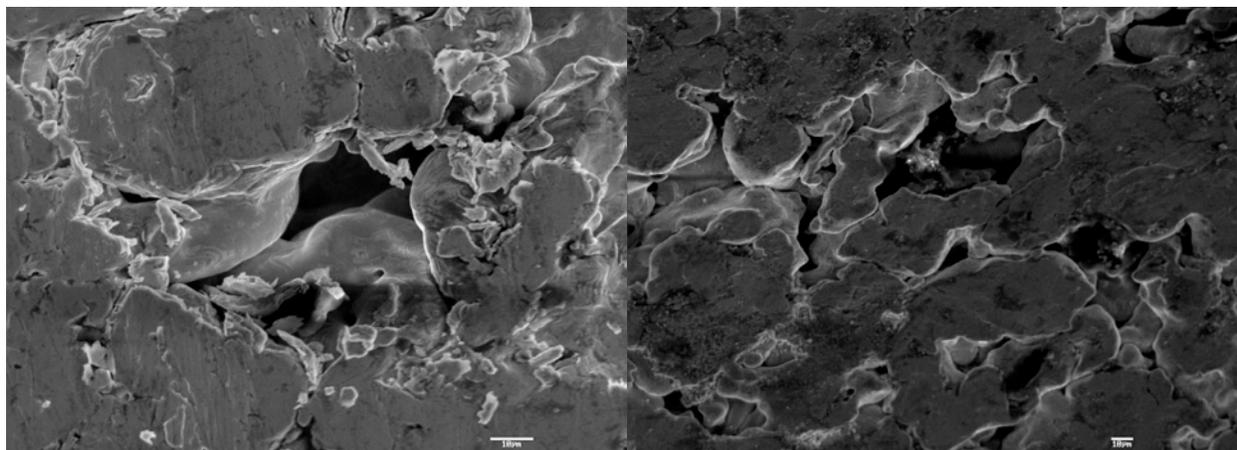
unused

subcritical

supercritical

Figure 4

With the SEM method, we also analyzed the membrane with the pore size of 1 μm . Here, too, there is no significant change in structure. The results are shown in Figure 5.



unused

supercritical

Figure 5

4. Conclusions

We have developed a setup for testing membranes under supercritical water conditions and have analyzed several stainless-steel filters. The structure and the texture of these filters did not change significantly. It seems that the elemental composition of elements in these filters is suitable for designing membranes for supercritical water. The next steps in researching membranes will be:

- finding more types of membranes to test
- extension of the test duration
- injection of corrosion particles to see the filtration potential of the membranes
- identifying and analyzing equipment which will resist high pressures and temperatures
- examining low volume systems and changing to fast volume systems

Acknowledgements

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