Oxide Dispersion Strengthened Steel Process Development at CANMET – MTL

by

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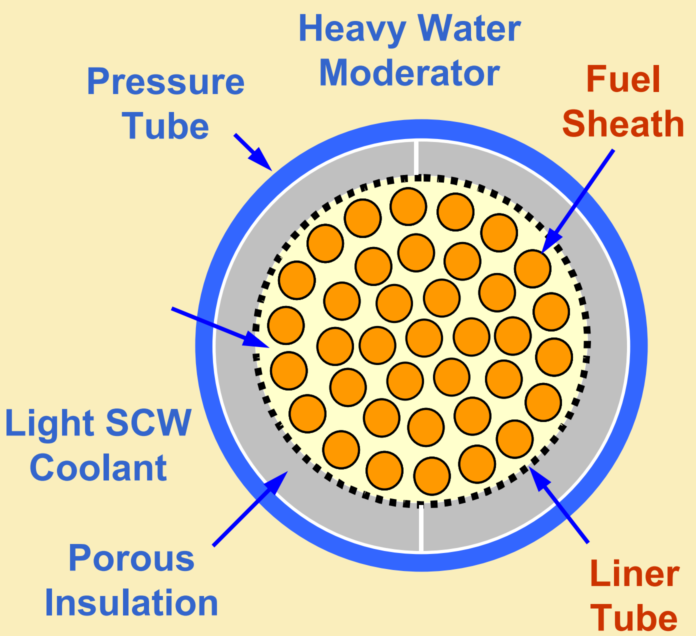
Outline

- Background
- Facility Development
- Preliminary Work
- Collaborations

Background

- Canada signed for Gen-IV Int. Forum for contributions in supercritical water-cooled (SWCR), and very high temperature reactors (VHTR).
- Material requirements for in-core structural components of SWCR:
  - Radiation-resistance against swelling
  - Corrosion-resistance in supercritical water environment
  - Creep-resistance up to 825°C
- Fe-Cr steels strengthened by nano-sized oxide particles is considered to be a viable solution.

SCWCR / HEC Design



Planned Processing Route

- Make up steels in MTL’s VIM furnace
- Atomise steels to powder: contract out
- Mechanically alloy steel powders with titanium and nano-sized yttria powder: procure and set up an attrition mill
- Transfer milled powder to a steel can for hot extrusion: refurbish an existing glove box with argon purifiers
- De-gas steel at 400°C, 0.1 Pa vacuum
- Crimp tubing to steel can, age powder at 900-1100°C
- Reheat to 1200°C and extrude to 1” dia. rod

Quality Assurance and Further Processing

- Check for oxide dispersion: micro-characterization, atom probe tomography at McMaster Univ.
- Mechanical properties: new creep machines
- Corrosion resistance: supercritical water loop
- Coat steels for corrosion-resistance / coatability
- Radiation resistance: collaboration with a neutron / proton irradiation facility
- Check hot and cold formability to tube
  - Pilger mill
- Investigate for appropriate welding methods / weldability
  - Friction stir welding / brazing

Metal Powder Production

Options available for atomization:

▪ Water-jet	Carl Blais, Laval Univ.
▪ Nitrogen gas	ASL, Sheffield, UK
▪ Argon gas	ATI Powder Metals
▪ Impulse	Hani Henein, Univ. of Calgary

Argon gas atomisation is the preferred method but expensive. Proceed with nitrogen gas atomisation.

Process coating steel by water-jet and argon gas atomisation.

Attrition Mill for Mechanical Alloying

Two potential suppliers have been identified:

- Union Process, Akron, OH
  - Lab Attritor 1-SD
  - 3 kg/batch powder processing capacity
  - Used by Japanese researchers
- Zoz GmbH, Wenden, Germany
  - Simolayer CM01, CM08 or CM20
  - 0.25 to 4 kg powder proc. capacity
  - Expensive

Attrition Mill Procurement

- Operation under a hydrogen atmosphere is desirable as argon contributes to radiation damage / swelling
- However, suppliers were unwilling to provide such a mill because of its safety concerns and difficulties in meeting the related codes.
- We made a decision to buy a mill for operation under argon and retrofit it later for hydrogen operation.

Glove Box

- Milled material surface is highly active, needs to be protected from oxygen during transfer to steel cans.
- Refurbish an existing glove box with oxygen and moisture purifiers for argon atmosphere with <1 ppm O_2 and H_2O.
  - Attach a controller and respective analyzers if feasable.

Vacuum De-gassing

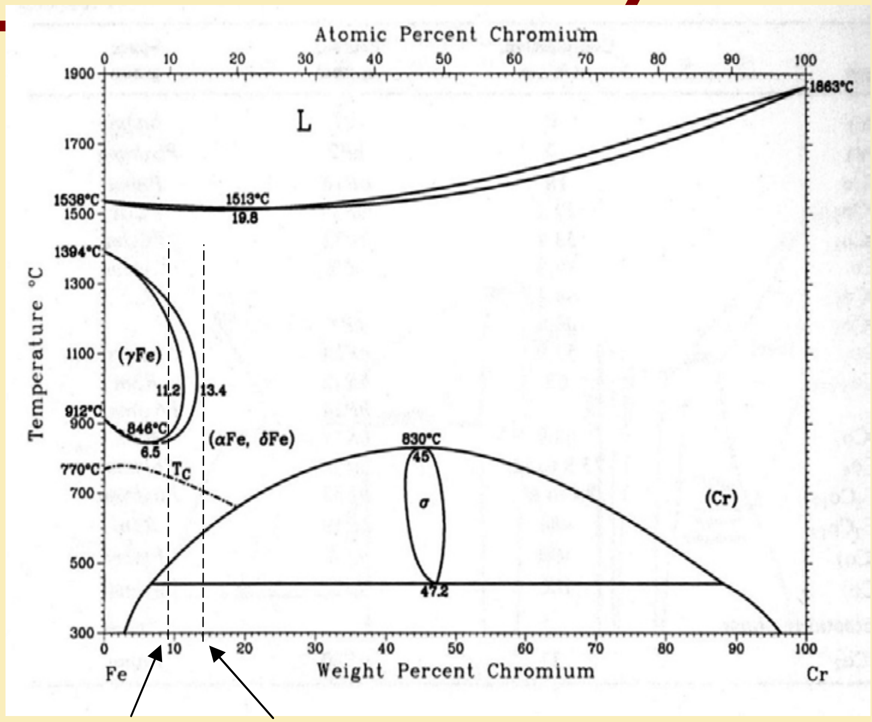
- Powders contained in steel cans and connected to crimpable tubes are degassed at 400°C.
- The following hardware was put together:
  - A vertical tube furnace with a 10” heated zone
  - Turbomolecular pump with a rotary vane backing pump
  - A two-stage high vacuum pump in parallel
  - Valves, gauges, tubing and connectors
- During a degassing operation, operate the vacuum pump for 2 h or until 1 Pa, and use the turbo-pump for final removal of volatiles only.

Crimpable Tubing

- Critical for retaining vacuum in steel can after degassing.
- Traditionally, copper tubing is used to hermetically seal freon in A/Cs. However, 1200°C reheat requires a higher temp. tubing metal.
- We tried a ¼" OD, cold-drawn low-C steel tubing in the following conditions: as-is, cleaned, annealed and cleaned, locally heated where crimping was to be applied.
  - Hermetic seals were achieved only 1 out of 3 cleaned tubing. Heating the tube prior to crimping did not help.
  - Fcc metals are know to crimp well. Try Ni-200 tubing next.

Steels Selected for Study

Steel	Nominal Composition
Base compositions for ODS	
MA957	14 Cr, 0.3 Mo
14WYT	14 Cr, 2 W
F/M steel	9 Cr, 0.12 C, 2 W
Coating steel	
Hi-Cr ferritic stainless	25 Cr, 0.8 Si



Fe, 9Cr   Fe, 14Cr

- Minimize common residuals (Si, Mn) to prevent He-swelling.
- No Al-killing for processing by atomisation.
- W is better for swelling but more of it is required for strengthening.
- F/M steel contains C for strengthening.

Steels Made in MTL

Steel	Heat No.	Cr	Mo	W	C	Si	Mn	Ni
Base compositions for ODS								
MA957	V9011	14.4	0.30	-	0.021	<0.01	0.076	<0.01
14WYT	V0003	14.4	-	2.1	0.017	0.027	<0.05	0.37
F/M steel	V0004	11.9	-	2.1	0.120	0.024	<0.05	0.33
Coating steel								
Hi-Cr ferritic	V0002	25.3	-	-	0.018	0.72	<0.05	<0.01

- Steels are vacuum induction melted and poured into cast iron molds
  - 225 kg steel melts, producing 4 x 50 kg ingots
  - Facility for on-line analysis and trim additions
- Ni contamination from FeW

Water-jet Atomisation

Steel	Comments	Yield (pct)	N (wt pct)	O (wt pct)	$d_{50\%}$ (μm)	t_{Cr2O3} (μm)
Fe-25Cr (V9012)	MTL Melt		0.012	0.024		
	MTL Ingot		0.016	0.045		
	Powder, water jet atom. at Laval Un.	44 / 30	**0.015**	**0.734**	70	0.40
	Powder, water jet atom. at ASL	45 / 35	**0.028**	**0.431**	30	0.10

Chromite film thickness of powders is based on mass balance for O-content:

$$t_{Cr2O3} = \frac{MW_{Cr2O3}}{3\ MW_O} \frac{\rho_{Fe}}{\rho_{Cr2O3}} \frac{\bar{d}_{50\%}}{600} (\text{wt\% O})$$

N solubility in Fe with Cr (wt %)		
Cr	N in Liq-Fe (1600°C)	N in α-Fe (1520°C)
0	0.045	0.013
9	0.110	*0.032*
14	0.180	*0.052*
25	0.420	*0.121*

Nitrogen Gas Atomisation

- Four steels were atomised at ASL, Sheffield, UK, Mar-10.
- Steels were remelted in an air induction furnace, and poured through a tundish fitted with a 5 mm dia. nozzle.
- Low Si content of the base steels resulted in poor fluidity, early blockages and poor yield:

Steel	Melt (kg)	Powder (kg)	Yield (pct.)	Middle fraction (-150/+40 μm, pct.)	APS (μm)
Base compositions for ODS					
MA957	173	30.8	18	50	55
14WYT	184	34.0	18	49	53
F/M steel	189	42.0	22	51	75
Coating steel					
Hi-Cr ferritic	187	180	96	47	80

Nitrogen Gas Atomisation

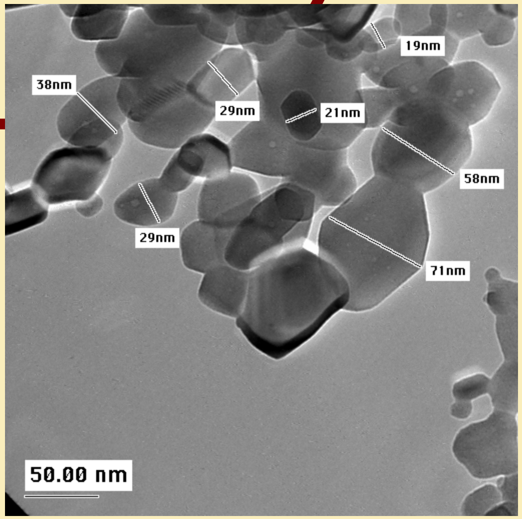
Steel	Comments	N (wt pct)	O (wt pct)	$d_{50\%}$ (μm)	t_{Cr2O3} (μm)
MA957	MTL Melt	0.012	0.017		
	ASL Melt	0.023	0.088		
	Powder, N2 gas atom. at ASL	**0.026**	**0.257**	55	0.11
14WYT	MTL Melt	0.016	0.037		
	ASL Melt	0.032	0.071		
	Powder, N2 gas atom. at ASL	**0.030**	**0.097**	53	0.04
F/M steel	MTL Melt	0.017	0.038		
	ASL Melt	0.029	0.062		
	Powder, N2 gas atom. at ASL	**0.031**	**0.079**	75	0.05
Hi-Cr	MTL Melt	0.021	0.025		
	ASL Melt	0.025	0.045		
	Powder, N2 gas atom. at ASL	**0.026**	**0.063**	80	0.04

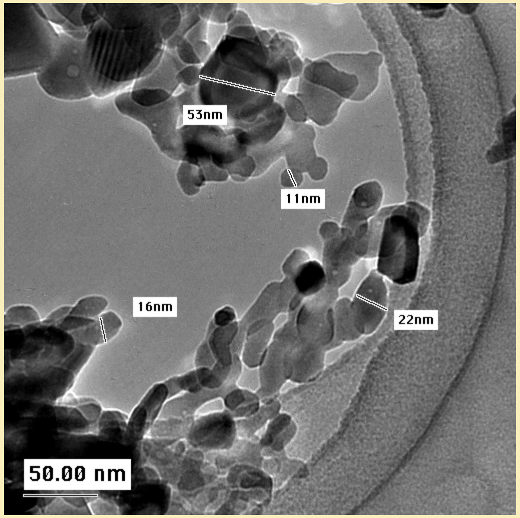
Nitrogen Uptake in Steels

- Nitrogen content of powder is 0.03 wt% or less.
- Ti:N mass ratio in TiN is 3.4:1
- Expect only 0.10% Ti to form TiN.
- Titanium is added to attrition mill at 0.3 – 0.7% of steel powder. Thus, sufficient excess titanium exists to form yttrium titanate for dispersion strengthening.

Other Raw Materials

- Yttria powder under 30 nm was only available from China. We bought a sample quantity from TJTM Inc. and checked its particle size under TEM.
  APS: 20-40 nm.
- Low-oxygen titanium powder was procured from Advanced Powders and Coatings, Quebec.
  Particle size: +45 / -106 µm.
  (0.090% O, 0.003% N by MTL's Leco)





Yttria powder from China

Collaborations

- Prof. Ge's group at Inst. of Nuclear Mats, Univ. of S&T, Beijing
  - Received ODS steels from China for creep testing
- Oak Ridge National Laboratory, TN
  - CANMET will arrange for nitrogen gas-atomised steel powders to be sent to ORNL for attrition milling