Procedure

The primary task of these experiments was to determine whether or not the sample tubes from MIR were fully inserted into the furnace during their processing. As it was not possible to glean any information from the coated length of tube, the only direct way to identify any temperature gradient is by comparing the oxides on the endcap and the "neck" of the sample. An additional benefit of this method is that identifying the temperature at the endcap of the sample – which was definitely in the furnace – confirms the processing temperature.

The review of research into the high-temperature oxidation of metals summarised above indicated that a characterization of the thickness of the oxide thin film on the uncoated stainless components of the sample tube would be the best way to proceed.

This determination could be more accurately performed by microscopic methods, either using scanning Auger electron spectroscopy to sputter away the oxide until metal is detected, or by using scanning electron microscopy on a heavily deformed sample to measure the thickness of the oxide at a crack.

Because of the large number of tubes to be considered, it was decided to use the faster, cheaper photometric method made possible by the interference tints discussed above. It was initially hoped that the photometer at the Alcan Research Laboratories in Kingston could be used to analyse the reflected wavelengths, but it has an incident beam about 3 cm in diameter – much too large for the small areas on the sample tube.

A series of small 321 stainless steel rods had one end machined to match the morphology of the flight specimen endcaps. These were then polished to 600 grit and cleaned with acetone to ensure that the surface conditions – so vital to the short-term formation of oxides – were as similar to those on the actual sample tubes as possible. These slugs were heated to a variety of temperatures for several different lengths of time, as follows:
First, they were processed for 30, 60 and 90 minutes at each of 300, 450, 550, and 700°C. It was noted from these runs that there is no variation in the interference tints with time for a given processing temperature. Therefore, just one 60 minute run was performed at each of 250, 350, 400, 500, 600, and 650°C, giving a spectrum of oxide samples from 250 to 700 °C at 50° intervals.

Since it was not possible to use a photometer to quantitatively determine the wavelengths that the oxide reflects, the only method which remained was visual comparison to the oxide colour/temperature standards discussed above. In order to minimise the effect that a narrow range of incident wavelength might have, the comparisons were made under conditions of indirect sunlight.

Two comparisons to the standards were performed on each sample tube: the endcap, and the hollow "neck" of the tube, just above the short plug section. While it was generally not possible to identify an oxide colour corresponding to an exact temperature, it was not difficult to "bracket" the flight sample in a 50° range between two standards.