Protocol for Characterization of Thermo-mechanical Properties

Sample description:

The thermo-mechanical properties of SOFC materials were measured by 4-point bending (strength, elastic and creep properties) and dilatometry (thermal expansion).

The tested Ni-YSZ material corresponds to the standard SOLIDpower anode-supported cells (Mezzolombardo, Italy). NiO-YSZ samples produced from the same raw materials and by the same procedures as the commercial cells were provided by SOLIDpower for testing. The only difference was the absence of the co-cast electrolyte layer. Measurements were performed in both oxidized and reduced state. In the latter case, the samples were reduced directly in the four-point bending test setup at a temperature of 800°C. The edges of the samples for strength measurements were inspected by optical microscopy and grinded until a sufficient quality was obtained.

The fabrication of the glass-ceramic samples for testing in four-point bending proved difficult and required several attempts and adjustments of the fabrication procedure. The processing route comprises:

- The deposition of the glass paste onto a compliant support.
- The heat-up to the sintering temperature of the glass in air.
- Devitrification by exposure to a fixed temperature.
- Cool down to room temperature.
- Removal of the support.
- Cutting of the plate to obtain samples of the adequate dimensions.
- Grinding to guarantee a uniform sample thickness.

The sintered parts typically have wavy surfaces, because a fully uniform shrinkage cannot be guaranteed. The ceramic paste was sintered into compliant moulds made of either thin metallic sheets or mica foils. Flat samples could not be produced directly, because the paste sticks to the vertical faces of the mould and the uniform shrinkage of the glass-ceramic material could not be guaranteed. Sample with adequate final dimensions were obtained by milling. Slabs with a dimension of 60 x 25 x 4 mm were first produced and then cut into samples with a dimension of 5 x 5 x 25 mm for dilatometry.

Thermo-mechanical measurements:

The coefficient of thermal expansion of the Ni(O)-YSZ and sealing materials was measured with a horizontal pushrod Netzsch DIL 402E dilatometer (Gerätebau GmbH, Selb, Germany). The alumina measurement setup containing the tested sample was enclosed into a gas-tight tube and heated-up by a tubular furnace. The expansion/shrinkage of the tested material during thermal cycling was measured by a linear variable displacement transformer (LVDT) which applied a constant contact force of 0.2 N on the sample throughout the testing. The temperature was monitored by a thermocouple placed at a distance of approximately of 1 mm from the sample. Considering the relatively small sample size and the mild heating ramps, the instantaneous temperature measured by the thermocouple was assumed close to that of the sample

Thermal expansion was measured on the length of rectangular-cuboid Ni(O)-YSZ samples with dimensions of 24.5 (length) x 5.5 (width) x 0.270 (thickness) mm. The samples were tested in forming gas (N_2:92% - H_2:8%) with a flow-rate of 3.5 l/min. The applied heating rate was 20°C/min from RT to 300°C and 10°C/min from 300 to 850°C, to increase the measurement accuracy by limiting the temperature inhomogeneity. The thermal expansion of the pristine and aged glass samples was measured following a similar procedure as the Ni-YSZ samples. It was measured on the length of rectangular-cuboid samples with dimensions of 25 (length) x 5 (width) x 5 (thickness) mm. The samples were tested in air and in forming gas (N_2:92% - H_2:8%) with a flow-rate of 3.5 l/min. The applied heating rate was 1°C/min from RT to 600°C.

Four-point bending mechanical testing was performed for the measurement of strength, elastic and creep properties. Two 4-point setup configurations were used. In both cases, the centre-to-centre distance of the outer rollers is 50 mm. The centre-to-centre distance of the inner rollers is 25 mm. The elastic properties and strength were measured in a setup with load control. The outer rollers are fixed on a static structure supported by two load cells. The inner rollers are fixed on a mobile frame, which is moved vertically at a constant rate by an electro-mechanic linear actuator. The elastic properties of reduced samples (Ni-YSZ) with dimensions of 60 (LT) x 25 (w) x 0.300 (h) mm were been measured at RT, 700, 750 and 800°C under reducing atmosphere and air at RT in oxidized state. The samples were heated at a rate of 150°C/h, followed by plateau during 8 h for stabilization. The displacement rate at the inner rollers has been controlled by the linear actuator at a rate of 0.1 mm/s. For strength measurements, samples (up to 30) were placed on the measurement slots and heated together up to the testing temperature (800°C), at a rate of 150°C/h, followed plateau during 8 h for stabilization. The samples were then loaded at a rate of 0.3 mm/s under fracture, one after the other. Because of the size of the furnace, a fully uniform temperature in the testing and storage regions cannot be guaranteed. Monitoring of the homogeneity of the temperature along the testing fixture show variations within 2°C, hence specific procedures were not required for temperature stabilization. Testing was performed in air and reducing atmosphere (N_2: 90% - H_2: 10%).