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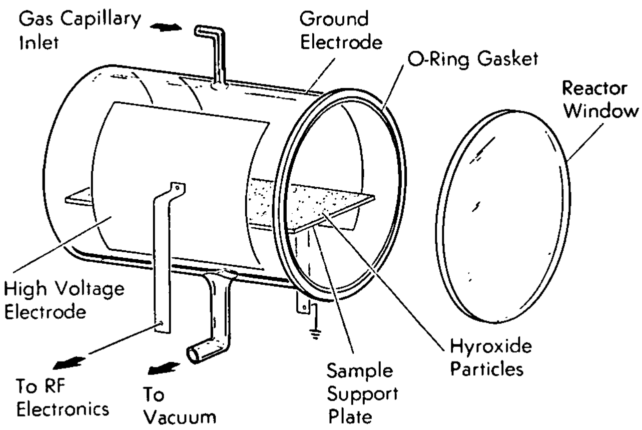
Oxygen Plasmas Used to Synthesize Superoxides

Alkali metal superoxides ordinarily are obtained by interaction of molecular oxygen with alkali metals or their salts; for example, sodium superoxide can be prepared by treating sodium hydroxide at 375°C with



oxygen at about 10 MN/m^2 (1500 psi). However, recent work has demonstrated that the synthesis of alkali metal superoxides can be more simply accomplished in oxygen plasmas created by radiofrequency (RF) discharges.

The apparatus used for study of the synthesis of superoxides is shown in the diagram. The reactor, made of borosilicate glass, is 76 mm in diameter and 150 mm long; stopcocks were located in the inlet and vacuum lines to permit isolation of the reactor volume from auxiliary apparatus (pumps, gas supplies, etc.). The electrodes were shaped to fit the sides of the reactor closely; one electrode was grounded, and the other was fed RF energy from a 13.56 MHz generator (100 watts) and an impedance matching network.

Alkali metal hydroxide particles were distributed uniformly over a 64-mm x 114-mm borosilicate plate in a dry box and the plate was put into place in the reactor; the reactor window was clamped in place (silicone O-ring) and then the assembled reactor was removed from the dry box and connected to the RF power system. The reactor was evacuated to a pressure of less than 0.13 N/m^2 (10^{-3} mm Hg) and then oxygen was allowed to enter via a flow meter; oxygen flow rates and pumping rates were manipulated to establish a desired flow rate and pressure in the reactor system. The RF plasma was initiated and maintained at a fixed power level. A typical oxygen flowrate was 50 cm^3 min^{-1} (S. T. P.) at a pressure of $\approx$133 N/m^2 (1.0 mm Hg); power inputs ordinarily were of the order of 70 watts.

After about 15 minutes of operation, the hydroxide particles appear to "melt" even though bulk temperature is not much higher than about 80°C. Gradually, the liquid phase disappears and there remains a dry film which becomes more yellow as the formation of superoxides proceeds (hydroxides and peroxides are white).

The conversion of rubidium and cesium hydroxides to superoxides (MO_2) by oxygen plasma discharge was studied in detail; the conversion of rubidium hydroxide as a function of time at different power levels and oxygen pressures was found to level off at about 58 percent for a typical particle size of starting material. It was noted that the reddish to dark orange ozonides of rubidium and cesium were formed during some syntheses, but these products are unstable and transitory.

(continued overleaf)

Notes:

1. Hydroxides could not be converted to superoxides in plasmas of water vapor or hydrogen peroxide.
2. Requests for further information may be directed to:

   Technology Utilization Officer
   Ames Research Center
   Moffett Field, California 94035
   Reference: TSP 72-10570

Patent status:

NASA has decided not to apply for a patent.

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