

had growth pits which resembled herring bone (Figure 15b). Some of the crystals were octahedra several tenths of a millimeter in size. Most, however, were of the order of 10 to 50 microns on an edge. X-ray diffraction of one of the octahedra showed it to be a single crystal. Electron diffraction showed the presence of a surface layer of nickel hydroxide resulting probably either from exposure to air or from water pickup during preparation for the diffraction studies.

Samples 27 and 22 pressed at 250,000 psi at temperatures of 1300° C and 1000° C respectively, showed Knoop hardness values of 728 and 800, respectively. Single crystal NiO has a hardness of 500 KHN.

c. Cr_2O_3

Six runs were made with Cr_2O_3 as shown in Table IX. In general, these did not yield as good specimens as had the other materials. The specimens were very friable and had low density. Often, upon opening the chamber a pronounced odor of H_2S was noticed. This indicated that the decomposition of the sulfate left sulfur as an impurity which reacted with the water from the pyrophyllite to give a vapor phase of H_2S during pressing. One specimen, however, pressed at 1400° C and 250,000 psi had a fair structure and gave a Knoop hardness of 1570 under a 500 gram load. Upon retest with a 100 gram load the hardness was 1100.

d. Al_2O_3

Linde B alumina is a mixture of γ -alumina, 0.02 micron grain size, and α -alumina, 0.20 micron grain size. The as-received material was preheated to 1000° C to drive off any volatiles and then isostatically cold pressed at approximately 20,000 psi and again at 38,000 psi to a green density of about 58 percent theoretical density.

The prepressed Linde B alumina was placed into the high-pressure apparatus with a Pt-Pt+10 percent Rh thermocouple, and pressed at 12.5 kilobars for approximately 30 minutes at temperatures of 500° to 1950° C.

The lower temperature experiments yielded a sample containing two distinct areas (Figure 16). The dark area formed almost a perfect cylinder inside of the deformed area. The deformed area next to the heater was white and porous as compared to the grey area which was more dense. The white area contained γ and α alumina. The grey contained only α -alumina. The higher temperature experiments conducted near 1000° C yielded samples which were very dense and had a Knoop hardness of 2200 to 2300 with a 100-gram load (Figure 17). The resulting grain size was approximately 1 micron for these conditions.

TABLE IX
Cr₂O₃ PREPARATIONS

Run No.	Pressure (psi)	Temperature (° C)	Power (watts)	Time (min)	Results
29	250,000	1230*	1584	6	Fair-good
28	250,000	(1260)*	1607	4	Fair
12	250,000	1400	1680	10	KHN 1571 (500) Fair structure
16	250,000	1580*	1776	3	
15	500,000	940*	1554	10	Very poor
30	500,000	1250	1757	4	Very poor

*Open thermocouple, tmeperature obtained in interpolation of power versus temperature calibration curves.