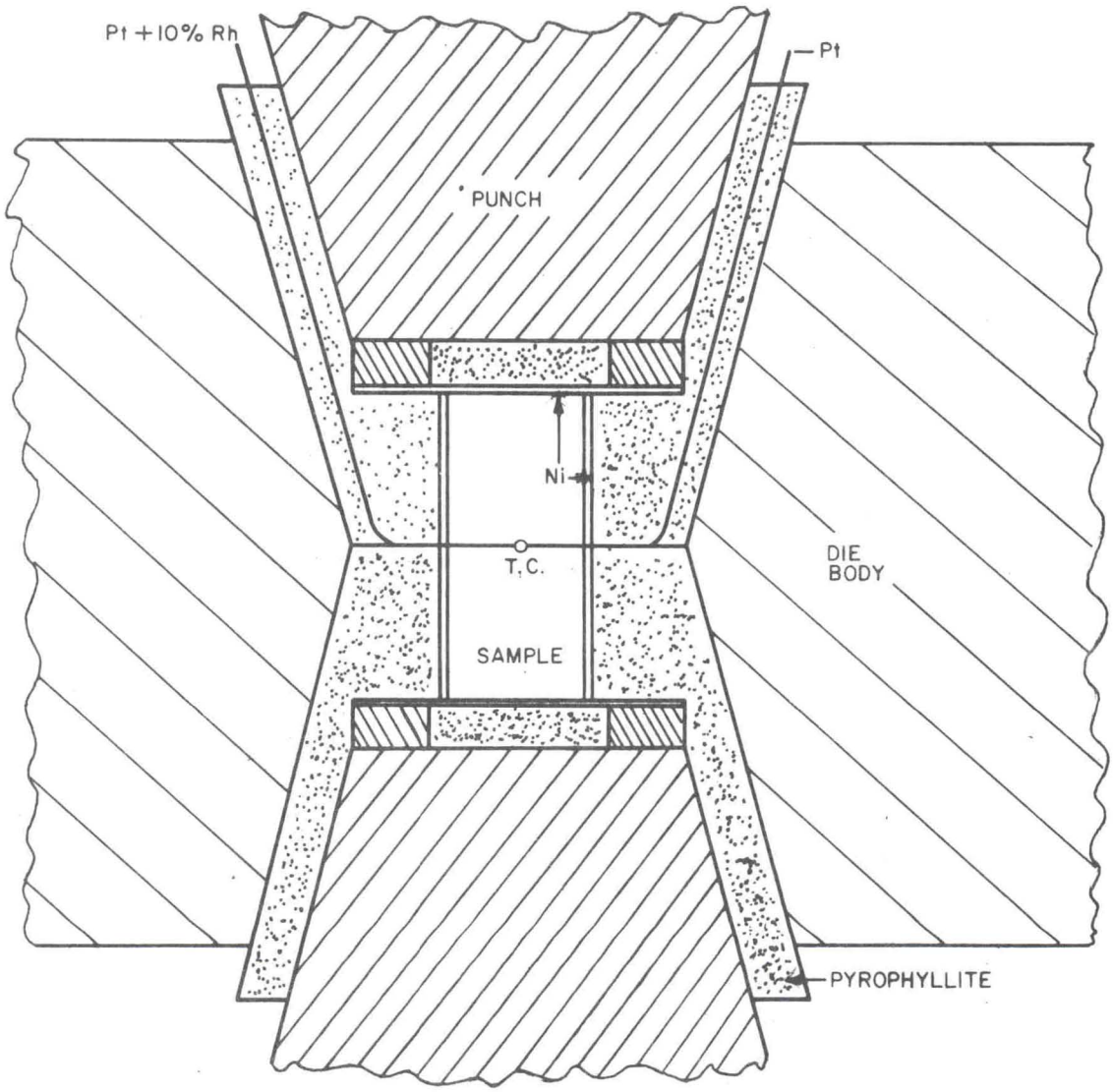

Figure 5 SAMPLE GEOMETRY FOR OXIDE DENSIFICATION STUDIES

The temperature observed was plotted against the power input. This curve gave a direct relationship of power versus temperature within 20° C, which was useful in the event the thermocouple opened during subsequent runs. In all cases where a thermocouple was used, a curve was obtained for the power input versus temperature.

3. Sample Design

The majority of experiments conducted in the 1 x 10^6 psi apparatus contained samples which were 0.500-inch in diameter by 1.000-inch long. The sample was placed inside of a tube which was either metal or graphite for resistance-heating purposes. The sample was enclosed by electrically conducting materials at each end of the sample. The remaining part of the sample was enclosed by pyrophyllite. The pyrophyllite, sometimes called "lava", is a fairly inert and stable material which acts as a pressure transmitting medium as well as an electrical insulator, and served as a gasket to maintain the sample under load. A schematic diagram of the sample is shown in Figure 5.

D. TECHNIQUES--TYPICAL RUN

A typical run containing MgO, NiO or Cr_2O_3 was set up as follows. The oxide was preheated to at least 600° C for 8 hours to drive off any volatiles that might be present. The powder was then placed into a polyvinyl chloride bag and pressed isostatically to 22,000 psi. The piece was removed, crushed, and prepressed at 35,000 psi. After removal from the isostatic press, it was then machined to fit the resistance heater. The sample was placed into the pyrophyllite gasket and loaded into the die body. With the previously aligned punches in place in the 400-ton press, the die was put in place on the lower punch. Thermocouple and strain gage loads were attached as required. Although the specific program varied with the sample and experiment, typically the sample was brought to the desired pressure by adjusting the press load to that indicated by the calibration runs. The temperature was then raised to the desired value by resistance heating of the metal or graphite sleeve. The attainment of the desired temperature was judged either by direct indication from internal thermocouples or by matching power-temperature curves with previous runs. In those runs, where new phases, possibly only metastable at lower temperature and pressure were sought, the sample was "quenched" by simultaneous removal of pressure and electrical power. In other cases where dense ceramic pieces were desired, a programed reduction of both pressure and temperature was followed to minimize mechanical and thermal shock.

Upon completion of the run, the sample along with the metal or graphite heater, was removed from the apparatus. The resistance heater was removed and the sample was exposed. The sample was examined by electron microscopy, X-ray diffraction or metallography.