Submitted to J. Sci. Instr. June 1962 -
I recommended no H. T. Hall July 6, 1962

MODIFIED "GIRDLE" HIGH-PRESSURE HIGH-TEMPERATURE DEVICE FOR PRESSURES TO 140 KILOBARS (KENNEDY SCALE)*

by

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(Received June 22, 1962

The "girdle" ultra-high-pressure device as originally described[1] called for either iron oxide powder or premachined compressible gaskets for insulation between the pistons and die. The effect of gaskets on the efficiency and ultimate sample pressure attainable in the girdle was not discussed. An apparatus using compressible gaskets, described by Daniels and Jones[2], was similar to the girdle. The highest pressure mentioned in the description of the Daniel's device was 100,000 kg/cm^2 on the Bridgman scale or approximately 75 kb on the Kennedy scale[3].

It has been possible by use of special gype gaskets to obtain sample pressures to about 140 kb in a modified girdle apparatus. At about 120 kb and 1200 C, stishovite[4, 5], the rutile-type high-pressure modification of SiO_2, was produced in this apparatus.

The piston-die-binding ring assembly in the revised girdle is essentially similar to that described previously[1]. The truncated cone portion of the pistons has a 35° cone angle and 1/2-inch piston face. The die is made with a 35° cone angle, 1/2-inch-bore diameter and 1/4-inch-bore height. Tungsten carbide with 6 per cent cobalt binder is used for the pistons. The die is made of Carpenter Hampden steel hardened to Rockwell C 60-63. The use of an expandable steel die instead of a tungsten carbide die is an important feature of the girdle apparatus.

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* Supported by Atomic Energy Commission.

The changes resulting in increased efficiency and higher ultimate sample pressure in the girdle modification are in the specimen cell and the gasket configuration. A schematic of the specimen cell and gaskets used in synthesizing stishovite are shown in Figure 1. An essential part of the assembly is the polyethylene films separating the gaskets from the die and piston. The films consist of ~~0.0004~~ .004-inch polyethylene sheets precut and joined at the edge by pressure and heat to form cones.

In synthesizing stishovite, a 1/16-inch I.D. platinum tube with 0.005-inch wall thickness was used both as container and heater. Electrical contact was made on either end to the piston via a 0.005-inch platinum disk, a steel washer with pyrophyllite insert and steel disk as shown in Figure 1. The heater requirements for this synthesis were 675 amperes and 0.92 volts, and the press load was 300 tons.

For pressure calibration, a cylinder of silver chloride enclosing an axial metal wire was substituted for the platinum tube. Resistance versus pressure was determined for bismuth, iron and barium. The bismuth VI-VII transition occurred at 210 tons press load. Sharp discontinuities were not observed in iron and barium above 200 tons press load. There was however the beginning of a rapid increase in resistance with pressure at about ~~210~~ 340 tons press load in barium and about ~~340~~ 310 tons in iron. If the Drickamer[6] values of 90 kb for the bismuth VI-VIII, 130 kb for iron and 144 kb for the upper barium transitions are used, the sample pressure required for stishovite synthesis was about 120 kb. This agrees with Stishov's estimate if his figure of 160,000 atm is converted to the Kennedy scale.

Approximate sample temperature at high pressure is determined from heater wattage. For analytical purposes it is desirable to measure sample temperature at pressure. Work is in progress on methods of bringing out thermocouples at the highest sample pressures.

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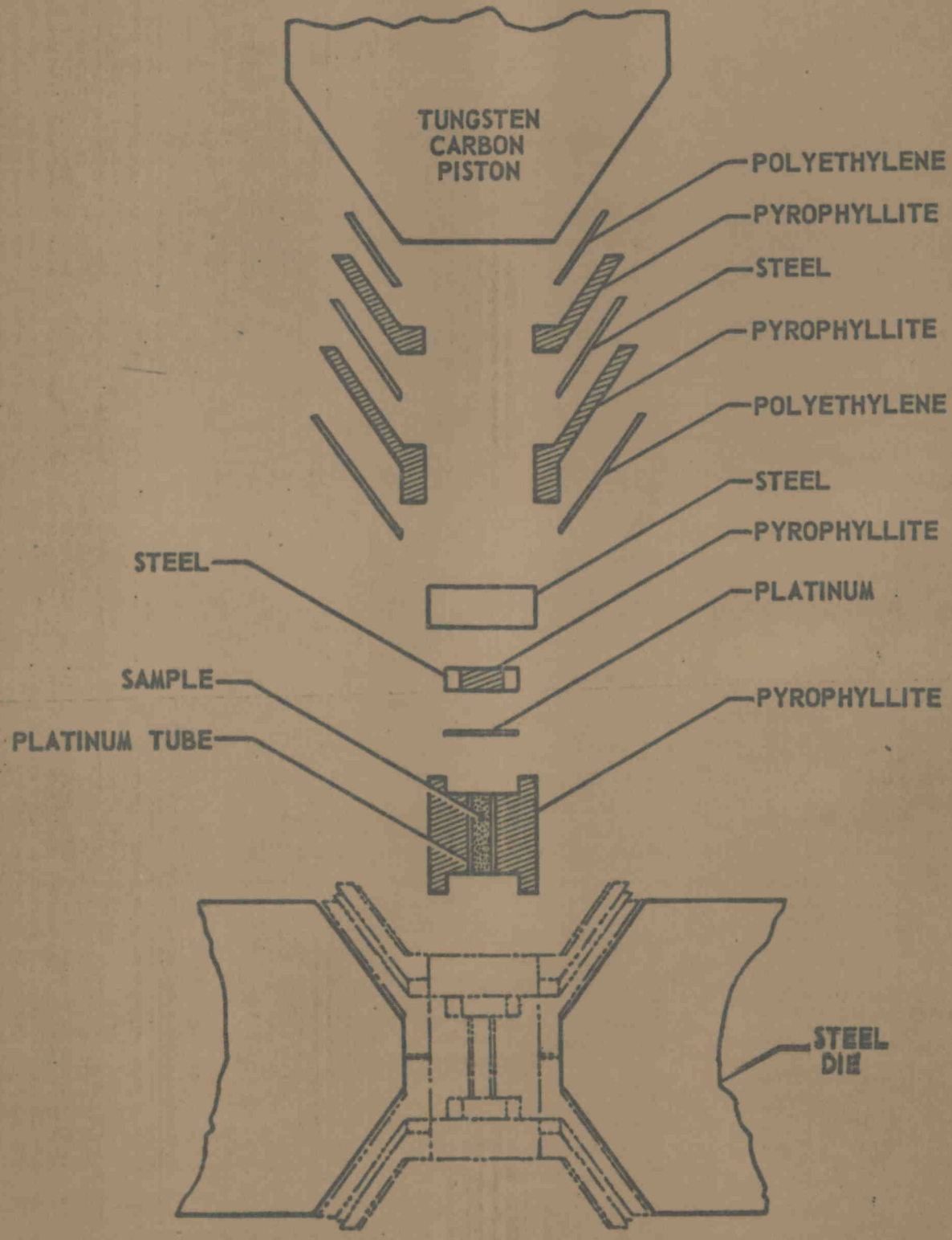
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