

The octahedral faces show striations parallel to the edges  $\langle 110 \rangle$ . Cleavage traces are noticed only in a few grains. The optical study shows light to dark green polarization colours and high relief, with well-developed fractures; the mineral is isotropic.

The cell-edge, calculated from X-ray powder data, is 8.098 Å, intermediate between those of gahnite ( $a$  8.062 Å) and hercynite ( $a$  8.119 Å).

Well-developed crystals of gahnite, collected by hand picking, were analysed by standard wet chemical methods after sodium peroxide attack. The chemical analysis of the mineral gives: SiO<sub>2</sub> 1.10 %, Al<sub>2</sub>O<sub>3</sub> 57.86 %, FeO (total iron expressed as FeO) 7.79 %, MnO 0.50 %, ZnO 32.50 %, total 99.75 %. The atomic ratios calculated to 32 oxygen are: Zn 5.69; Fe<sup>2+</sup> 1.53; Mn 0.09; Al 15.90. From the chemical analysis, the mineral could be called ferroan gahnite. Further work (by way of chemical analysis of more samples and the determination of trace element content of gahnite) is in progress and will be reported elsewhere.

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## A modified closure for high-pressure cold-seal pressure vessels

IN 1963 Luth and Tuttle (1963) described modifications of the original Tuttle (1949) cold-seal pressure vessel that enabled the useful working limits of the apparatus to be extended to approximately 11 kb and 750 °C. The essence of this modification was the transmission of pressurized fluid (argon) through  $\frac{3}{16}$ -inch O.D. tubing to a hardened cone that was forced into the pressure vessel by a thrust washer and closure nut. Lubrication of the closure nut and thrust washer were recommended to prevent rotation of the cone and consequent galling in its seat on tightening.

This type of vessel has been manufactured for some years now by the Tem Press Co.<sup>1</sup> and we have used their product with considerable success. Our experience in using

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the vessels for some four years has suggested to us an alternative closure, not in itself unique but not, to our knowledge, normally used for the 'test-tube' type of cold seal pressure vessel. The prime reason for seeking an alternative closure was the damage inflicted on both the cone and the vessel itself in attempting to seal slow gas leaks in the system. Our experience with various lubricants on the thrust washer suggests that the cone does in fact rotate at high torque rates (in excess of about 90 ft lb.).

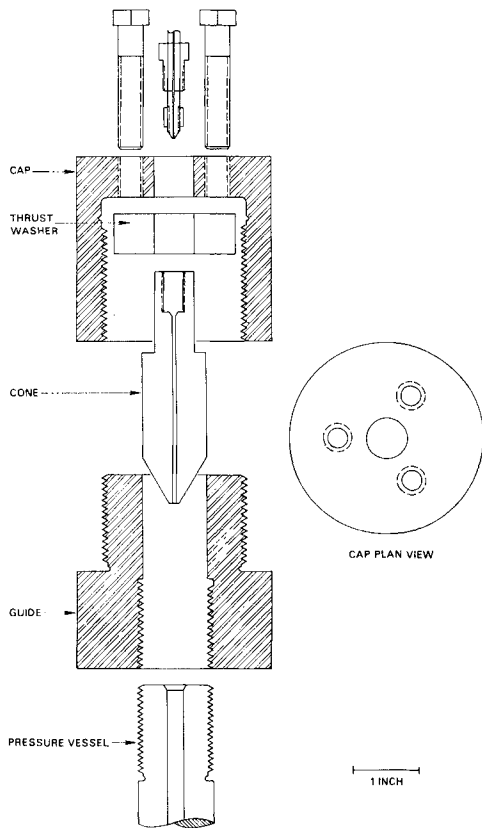


FIG. 1. 'Exploded' view of the closure components. The guide and cap are hand tightened and the cone sealed into the pressure vessel by tightening bolts through the three holes in the cap.

closure cone. The guide piece for the cone is threaded directly on to the pressure vessel but is only hand tightened and its inside diameter is carefully matched with the cone to achieve alignment of the male and female cones. In the first few specimens of this closure little attention was paid to the tolerance between these two components and a number of slow leaks occurred, that could be traced only to this cone seal.

The slight misalignment of the  $59^\circ$  male cone with the  $60^\circ$  female cone was sufficient to allow a slow leak to develop. With a close fit between the cone and its guide it is

The closure described by Luth and Tuttle has given good results with newly machined parts but only limited success has been achieved on repeated use of components. Gas leaks we have encountered may be insignificant for experiments of one or two days' duration but we have endeavoured to make the pressure system gas tight for experiments of up to three months' duration. One surprising point is the variable performance of apparently identical closures. Occasionally a pressure vessel and closure cone have been used successfully for six or eight experiments at pressures up to 9 kb. Others, however, machined from the same material and finished to the same specifications have failed to give a satisfactory seal after a single experiment. In each such case the cause of the leak could be traced to deformation of either the cone or the vessel or both.

In order to remove any possibility of movement of the cone against the pressure vessel we have adopted the design shown in fig. 1. The thrust of the cone into the vessel is made by tightening the three high tensile bolts through the cap on to a hardened thrust washer placed at the back of the

not possible to misalign the cone on tightening the bolts. The outside diameter of the cone and the inside diameter of the guide are machined with the following tolerances: cone 1.0000 to 1.0002 in. outside diameter, guide 1.0003 to 1.0005 in. inside diameter. The cap is hand tightened on to the guide and sequential tightening of the three bolts, increasing the torque by about 2 ft. lb. each time, gives the best results. A maximum torque of 20 ft. lb. on each of the three bolts has proved adequate to make a seal with a gas pressure of 140 000 lb. in<sup>-2</sup>.

The various components of this closure have all been machined from hardened steel. Uddeholm Company's orvar steel has proved suitable for all four major components with the following Rockwell 'C' hardness: the guide and cap 48 to 50, the cone 45 to 47, and the thrust washer 52 to 54. Standard grade five tensile bolts  $\frac{3}{8}$  in., 16 N.C., were satisfactory after grinding a smooth flat end on each bolt.  $\frac{3}{16}$  in. tubing and Harwood<sup>1</sup> fittings are used to connect the tubing to the back of the cone.

Use of these closures for almost three years has greatly reduced the necessity for remachining both the pressure vessel cone and the closure cone. The life of the vessel is thus greatly increased and the system allows immediate reuse of the components.

Our initial experience with this type of system suggested that the vessels manufactured from Rene 41 usually failed by a relative slow and harmless swelling of the vessel opposite the hot spot of the furnace (Fawcett, 1963; Fawcett and Yoder, 1966). Recently, however, the failures have been of an explosive nature. Two vessels fractured longitudinally along half of their length during experiments that had run for about three weeks at 700 °C and 9 kb—half of their planned duration. No accurate record of bomb use had been maintained but each of these vessels had been in use for about 2½ years at pressures between 3 and 9 kb and temperatures between 400 °C and 800 °C.

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<sup>1</sup> Harwood Engineering Co., Walpole, Mass.