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A HIGH PRESSURE "CHECHEVITSA" (LENTIL)-TYPE MINI-CHAMBER MADE FROM SUPERHARD CERAMICS - BASED ON CUBIC BORON NITRIDE

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Abstract

This high-pressure chamber is based on the"anvil with recess" design - with a chamber diameter of 5 millimetres, and made from a superhard material based on cubic Boron Nitride. It has been specially developed to work in environments with pressures of up to 10 GPa.

Key words: *high-pressure device, Bridgman anvils, lentil-type high-pressure device, superhard materials, cubic boron nitride*

1. INTRODUCTION

In previous publications (A.A. Antanovich *et al*, 2011; A.A.Antanovich *et al*, 2012) we have described this superheat material based on cubic Boron Nitride - and its successful application in the production of high-pressure chambers, such as the Bridgman Anvil. Anvils with an operational area of 0.53 millimetres are capable of generating pressure up to levels of 40 GPA. This leaves us able to hope that this superhard material could be put to use for building high-pressure chambers of the slotted anvil, or "lentil" type (L.G.Khvostantsev *et al*, 2004), capable of withstanding lower pressures (up to 10 GPa) but with larger operational volume. This type of chamber would be in great demand for physics experiments involving high pressures and low temperatures – for example, for holding coil systems used when measuring the magnetic proclivity of samples.

2. THE EQUIPMENT AND RESULTS

A number of such "lentil"-style high-pressure chambers, whose operational components were manufactured from ceramics based on cubic Boron Nitride have been produced and put into use. The configuration, and operational volume of these chambers is detailed at Fig 1.

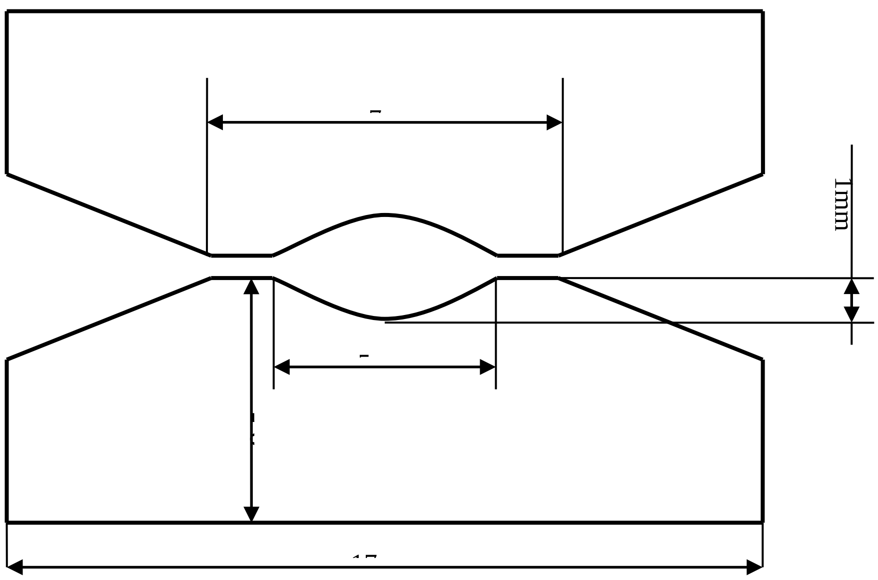

Fig 1

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The operational components of these chambers were then pressed into 35mm reinforcing steel rings. This permitted the chambers to be tested for use at low temperatures in automatic mode, by using a clamp mechanism of a fixed tensioning screw (see Fig 2) – which had previously been used (A.E.Petrova *et al* 2005) for miniature chambers of the 'toroid' set-up – manufactured from superhard carbide. This chamber was tested in actual experimental conditions to measure the specific heat of the compound $K_{0.3}MoO_3$.

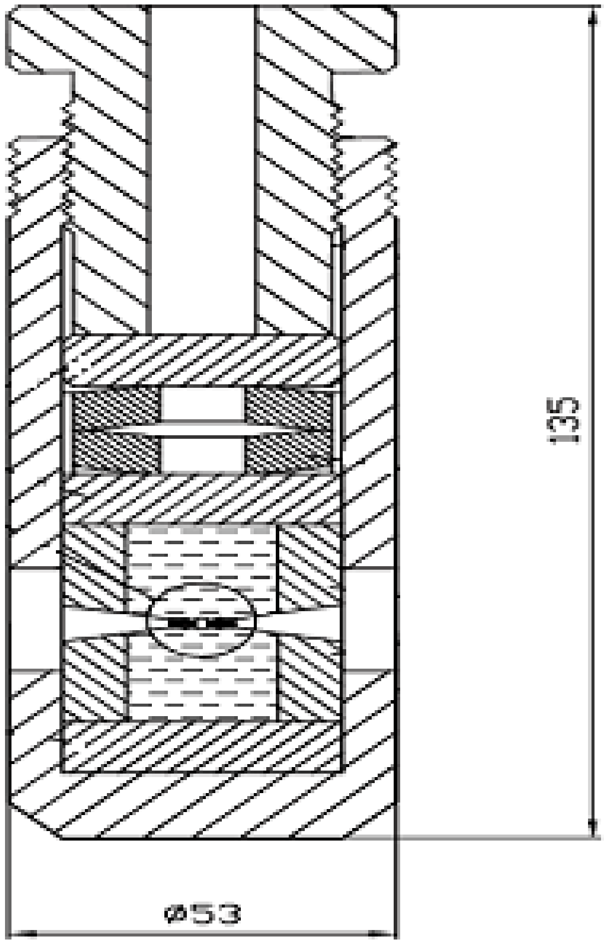


Fig 2

The chamber was filled by using a hydraulic press with a maximum capability of 25 tons, and then by the force on the chamber of the fixed tensioning screw – the chamber was taken out of the press, and put into a cryostat. The pressure was measured using a Manganin Gauge located in a teflon ampule filled with liquid. The ampule was placed in the centre of a gasket made from a mixture of aluminum oxide and epoxy resin. Eight wires were run through the ampule. Graphs illustrating the extent of resultant pressure occurring in the chamber appear at Fig 3.

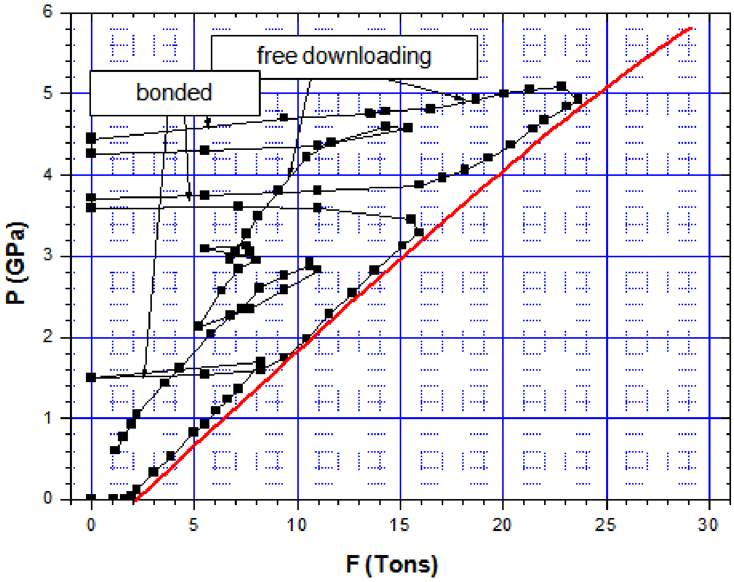


Fig 3

At the maximum force of 24 tons in the chamber the resultant pressure was 5 GPa. It must be noted that following a cooling cycle taking the temperature to that of liquid nitrogen, 77 Kelvin, after which the temperature was restored to room temperature with resulting load on the chamber, the pressure in the chamber reverted to the load-curve indicated in the diagram by the solid red line.

In the course of this experiment, readings were also taken on the influence of the temperature on the pressure inside the chamber. The dependence on temperature of the pressure in the chamber decreased relative to pressure at room temperature at different start-pressures, as shown in Fig 4.

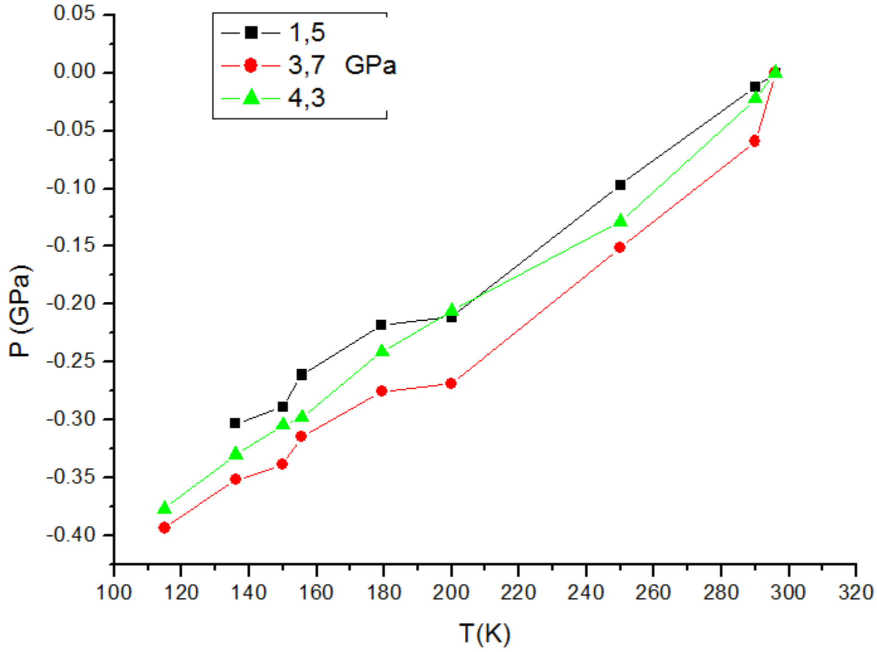


Fig 4

The pressure falls by around 0.4 GPa when cooled to the temperature of liquid nitrogen (77 Kelvin). This drop in pressure was calculated using a Manganin Gauge, bearing in mind the temperature dependency of its electrical resistance at zero pressure. The drop in pressure can be accounted for by the large coefficient of temperature expansion in the liquid in the ampule, when compared with temperature coefficient of expansion of the other components of the chamber.

No harm came to the chamber during or after the experiment. No faults or cracks to the surface of the chamber were detected when examined with an optical microscope, nor any signs of shape deformation. The appearance of the chamber after the experiment can be seen in Fig 5.

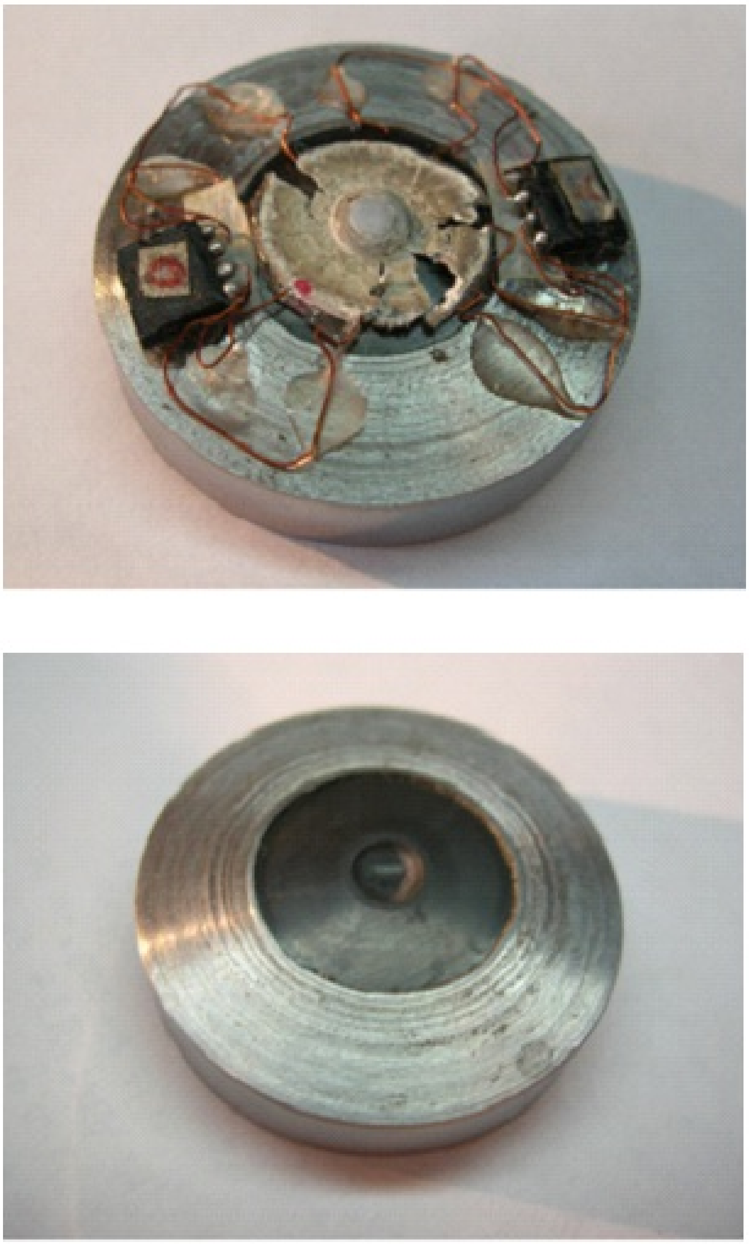
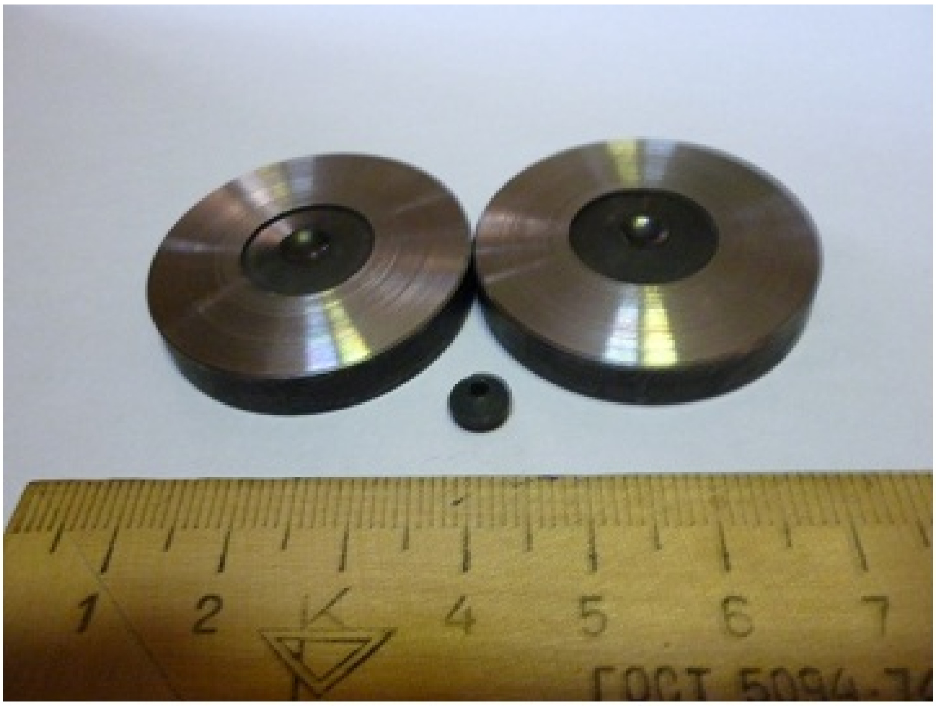


Fig 5

A further experiment with this high-pressure chamber involved subjecting it to a force of 50 tons with a hydraulic press. The Pressure Cell (Fig 6a) was meanwhile pressured with a mixture of 80% $CaCO_3$ and amorphous boron (20%). The chamber did not collapse as a result of the test (Fig 6b).



a

b

Fig 6

The pressure was not measured during this experiment, but was estimated to have been not lower than 10 GPa.

3. CONCLUSIONS

The superhard ceramic - produced by a method of impregnating cubic Boron Nitride with grains of aluminum at high pressure and high temperature – demonstrated its operational potential under two differing extreme conditions. The first was withstanding pressures of up to 40 GPa with a limited operational volume. The second was withstanding pressure up to 10 GPa with a large operational volume. The next stage will involve creating pressure chambers extending to 15 GPa and an operational volume of several cubic millimetres – vital for measuring numerous physical properties of materials exposed to low temperatures and pressures greater than 10 GPa.

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