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Multianvil calibration and education: A four probe method to measure the entire force-versus-pressure curve in a single run – performed as an interdisciplinary lab-course for students

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Abstract. The setup for and first experiences and results with a newly established lab-course on multianvil pressure calibration is reported. In order to provide a challenging and multidisciplinary task, as well as motivating results and an acceptable time scale, an assembly containing four different calibrants with consecutive phase transformations was adopted, thus making it possible for each group (3–4 students) to measure the entire pressure-versus-force range of the press in a single run. The course was performed by students of the subject “applied natural sciences” in their seventh term.

1. Introduction

A multianvil press (MAP) is usually regarded as a scientific instrument, used for institutional or university-borne high pressure research in the fields of geo- and materials sciences – or for the commercial synthesis of superhard materials such as diamond and *c*-BN. Even at universities, a MAP is—at least in the vast majority of cases—used by professional researchers or for project work of graduate students. In order to attract and educate interested newcomers within this still emerging field, institutions offer extra-curricular multianvil lab courses and graduate programs (see e.g. [1, 2]) since a long time. Additionally, the author is aware of at least three sites offering *curricular* activities that include usage of multianvil technique, in Germany [1, 3] and Austria [4]—a thorough world-wide research would certainly show many more. These courses are mostly addressed to students within the field of geosciences, solid state- or inorganic chemistry and deal with syntheses or phase conversions at high-pressure–high-temperature (HPHT) conditions which are the most common routine experiments carried out in large volume presses.

In this contribution, in contrary, the *calibration* of a high pressure multianvil apparatus at room temperature is presented as a multidisciplinary and versatile experiment. It can be adjusted to address graduate, as well as undergraduate students in many (more) and less specific subjects, such as physics, mechanical and electrical engineering, materials sciences, applied natural sciences etc. Its versatility stems from the fact that high pressure calibration requires knowledge and skills in various fields, ranging from the mechanics of machine parts as well as the behavior of rigid and elastic-plastic solids, electronic properties of metals and semiconductors and their structural

phase transformations, electric circuitry and measurement procedures, geometry of polyhedra, and last but not least spatial sense and good deal of craftsmanship in handling tiny and delicate components. According to the background and interests of the respective student group, each of these aspects may be treated more—or less—intensively, just as required.

2. Experimental

2.1. General experimental setup and equipment

A uniaxial hydraulic laboratory press with a maximum hydraulic pressure of 700 bar, i.e. equivalent to a ram force of 10 MN (\cong 1000 metric tonnes), equipped with a two-stage split-cylinder ‘Walker-type’ [5] multianvil module (hereafter named ‘Walker-module’) was used. Both components were provided by Vötsch Maschinenbau GmbH, Mainleus, Germany.

The first stage anvils (‘wedges’) are made of hardened tool steel with a square face side of 60 mm towards the second stage. The second stage anvils consisted of 8 hard metal cubes (material: Toshiba Grade F or Ceratizit TSM 20) with 32 mm edge length and 5.0 mm corner truncations. Lubrication and electrical insulation of the upper wedges against the containment ring of the module was provided by a double-layer of polyacetate foil (OHP-foil) [5, 6]. Between the foils and on the bare lower wedges, a slurry of MoS₂ or hexagonal boron nitride powder in ethanol was applied by a brush and left to dry before assembly.

In all but one cases, the gaskets of the inner pressure assembly were made of natural pyrophyllite with 2.2 mm thickness¹. These were supported with glass-fibre reinforced teflon tape (Böhme Kunststofftechnik GmbH & Co. KG, Schwarzenbek, Germany) and 0.22 mm thick cardboard, each one glued to one anvil face opposite to each other and both cut back to the outer dimensions of the gaskets. Electrical insulation and mechanical buffering between first and second stage was provided as usual by glassfibre-epoxy sheets, 0.8 mm thick. In contrary to many other laboratories that use 6 large rectangular sheets for all 8 inner anvils however, in the present case, each anvil was provided with 3 small epoxy platelets that were glued put with the inner cube corners, in order to prevent intrusion of the material into the closing gaps between the anvils during compression. Those anvils that were in equatorial position had smaller epoxy sheets to leave space for the electrical leads, required for the resistance measurements (c.f. Fig. 1(b), left part). Copper tape with conducting adhesive as usually employed for scanning electron microscopy sample preparation was used for the electrical leads. For the axial anvils, copper foil strips were put through slits within the epoxy sheets, as is traditionally done to make contacts for sample heating. By this manner, all eight faces of the octahedral pressure cell were contacted through the corresponding anvils.

For the resistance data recording, a digital multimeter (Keithley, 2700 series) in conjunction with an in-house programmed LabViewTM script, and a personal computer were used (\rightarrow ‘R-Log’). The hydraulic pressure profile was programmed and logged on the same computer (\rightarrow ‘P-Log’), using the software of the manufacturer of the press.

In the present study, pressure media consisted of commercially available MgO:Cr octahedra with \sim 30% porosity and 10 mm nominal edge length² (Ceramic Substrates and Components Ltd., Isle of Wight, U.K.).

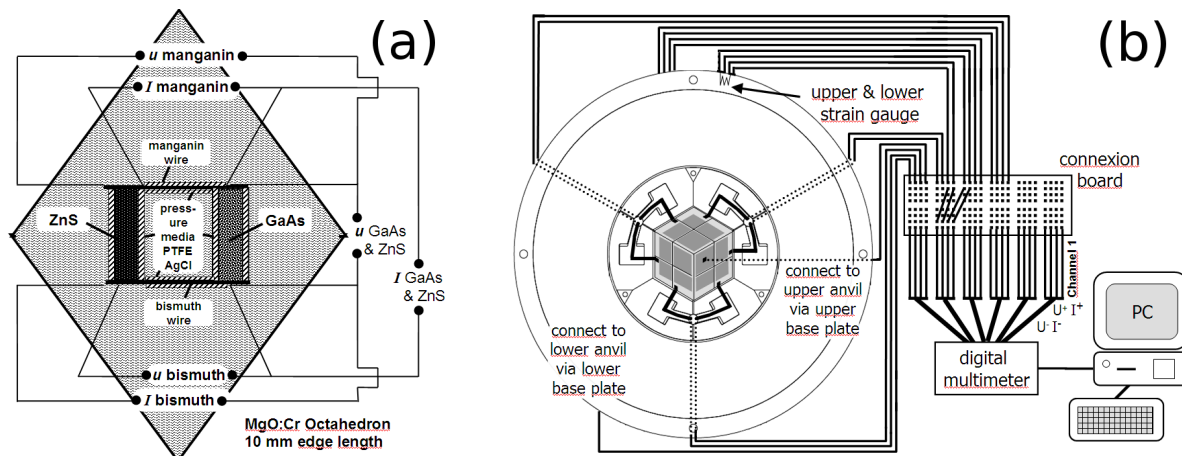
2.2. Specific setup, methods, materials and preparations for the lab-course

A scheme of the inner arrangement of the calibrants within the octahedron and the measurement circuits is shown in Figure 1 (a). In reality, the measurement circuits had to be established by appropriate partitioning of the eight octahedron faces (i.e. the eight electrically connected anvils) to the according measurement channels of the multimeter, as indicated in the schematic drawing in Fig. 1 (b).

¹ all other dimensions calculated according to the true octahedron edge length with program “Gasket” by Kurt Leinenweber [7]

² true edge lengths ranged between 9.7 and 9.85 mm

Figure 1: (a) Schematic cross section of the octahedral pressure cell; (b) Electrical connexion scheme of the experiment with top view into 'Walker module' (circular left feature), with the nested second stage anvils (tungsten carbide cubes, light grey) in the centre. The anvils have separate glassfibre-epoxy sheets (dark grey) of different sizes. The six equatorial cubes are contacted with adhesive copper leads, attached close to the edge of each cube. At the connexion board, the four-wire measurement of the upper strain gauge on Channel 5 is shown 'plugged-in' as an example. All other connexions are omitted for clarity.



A connexion board (plugboard) that is regularly used for building and testing electronic circuits and can be purchased in certain electronic stores proved to be extremely useful for this purpose, and a flexible means for realising different measurement setups in general. In the lab-course, the students will find an empty plugboard and have to establish all the connexions themselves. This is especially tricky with respect to different octahedron orientations (*vide infra*). In addition to the resistance measurement *inside* the pressure cell, the swelling of the containment ring during compression is measured by two strain gauges, as in Walker's original work [5].

In preparation of the lab course, for each group an octahedron was sectioned with a diamond-wire saw, so that two pyramid caps and a rectangular central platelet of ~ 2 mm thickness were created. The platelet was provided with two 1 mm bores, connected by ~ 0.4 mm deep grooves on both sides (Fig. 2 (a)).

In order to ensure a quick progress of the assembly of the calibrant octahedron during the lab course, the following further parts were pre-fabricated:

- GaAs: $2 \times 0.2 \times 0.5$ mm, semiconducting quality (Freiberg Compound Materials, 0.35 M Ω cm) cut from a wafer with diamond wire saw³
- ZnS: $2 \times 0.2 \times 0.5$ mm cut from large, amber coloured crystallites of a local zinc blende ore (diamond wire saw)
- Bi thin foil (< 0.1 mm) made by quickly squeezing a molten drop or a solid piece between metal faces
- pieces of manganin wire $\varnothing = 0.12$ mm $\ell = 4$ mm, 0.39 Ω /cm with silk and varnish insulation (Isabellenhütte, Dillenburg, Germany), used without any further thermal or mechanical seasoning [8]
- several pieces of Teflon tubing, $\varnothing_{out} = 1$ mm, $\ell = 1.7$ – 2 mm (OMEGA Engineering, INC., same material as used for thermocouple insulation)
- AgCl thin plate, pressed to ~ 0.5 mm with (stainless) steel die
- copper tape with conducting adhesive (Plano GmbH, Wetzlar, Germany) attached to a teflon block to be cut to shape with scalpel

³ caution! GaAs saw dust and slurry is toxic, water/liquids that were used for cutting require proper disposal!

The parts were stored in small dispenser boxes, placed close to the binocular microscope where the assembly was to be made during the course.

3. Scheduling of consecutive lab courses

According to the curriculum, the time available for the lab course was about half a day. In multianvil experiments, in order to prevent damage to the second stage anvils, the typical duration of a full compression-decompression cycle up to pressures of 20 GPa takes about 16 hours. Decompression is especially critical, because as the external force and hence also the confining internal pressure is released, strong tensile stresses can emerge in some regions of the brittle tungsten carbide cubes. Therefore, decompression is usually performed two- to three times slower (i.e. within ~ 12 h) in order to ensure a proper re-arrangement of the assembly and relaxation of the extreme internal stresses. Compression is usually performed within 3 to 4 hours. As pressure calibration data is always given for ascending pressure, the presence of the students during this compression stage appeared a reasonable timing for the course. However, keeping in mind the required manufacture of the pressure assembly and all other necessary preparation steps, especially when carried out by unexperienced persons, it became clear that one group could not *prepare and perform one and the same* calibration run, but the tasks for one experiment had to be partitioned between two consecutive groups, leading to the following (idealized) schedule (Table 1):

Table 1: Partitioning a calibration experiment among two consecutive groups of students.

Student group N		decompression over night	Student group $N + 1$	
<i>run No. k</i>	<i>run No. k + 1</i>		<i>run No. k + 1</i>	...
- (remove prev. experim.)			- (remove prev. experim.)	
- inserting in press			→ inserting in press	
- wire connections			...	
- start measurement				
↑	meanwhile:			
compression	- assembly of octahedron			
↓	- assembly of anvils →			

4. Introductory literature and questions for the students

The students are not provided with an elaborate lab exercise manual (which also helps to save some preparation time of the teacher), instead they are asked to familiarise themselves with the employed techniques from the following original research publications. These can be e.g. deposited for download at a non-public server:

- ***Multianvil-technique in general:***
N. Kawai, et al. [9]: “The Generation of Ultrahigh Hydrostatic Pressures by a Split Sphere Apparatus”
- ***‘Walker-type’ multianvil apparatus:***
D. Walker, et al. [5]: “Some simplifications to multianvil devices for high pressure experiments”
- ***Concept of pressure calibration by fixed point phase transitions:***
A. Onodera, et al. [10]: “Fixed points for pressure calibration above 100 kbars related to semiconductor-metal transitions”

- **Concept of pressure calibration by continuous resistivity change:**

N. Fujioka, et al. [8]: “Electrical resistance of Manganin under high static pressures”

optional additional literature:

- **More detailed information about certain peculiarities of the Walker-Module:**⁴

D. Walker [6]: “Lubrication, gasketing, and precision in multianvil experiments”

- **Crystallographical details about transformations in Bismuth (as one of the most prominent fixed-point calibrants):**

O. Degtyareva, et al. [11]: “High-pressure structural studies of group-15 elements”

Moreover, with respect to the given literature and to a previous lecture, following questions are given to the students for further familiarisation with the upcoming experiment and for self control how much they have understood about certain important aspects of the underlying physics and methodologies.

Be prepared to explain:

- (i) the meaning of ‘hydrostatic pressure’ and ‘general stress state’
- (ii) which properties an effective pressure medium should have
- (iii) the difference between four-wire and two-wire method in electrical resistivity measurements
- (iv) how many leads can be skipped, when the individual resistance of *three* samples, concentrated within a small volume, is to be measured by the four-wire method? Construct possible circuit configurations for (A): all samples are metal, and (B): one sample is initially a semiconductor.

5. The lab-course itself: Preparing and performing the experiment

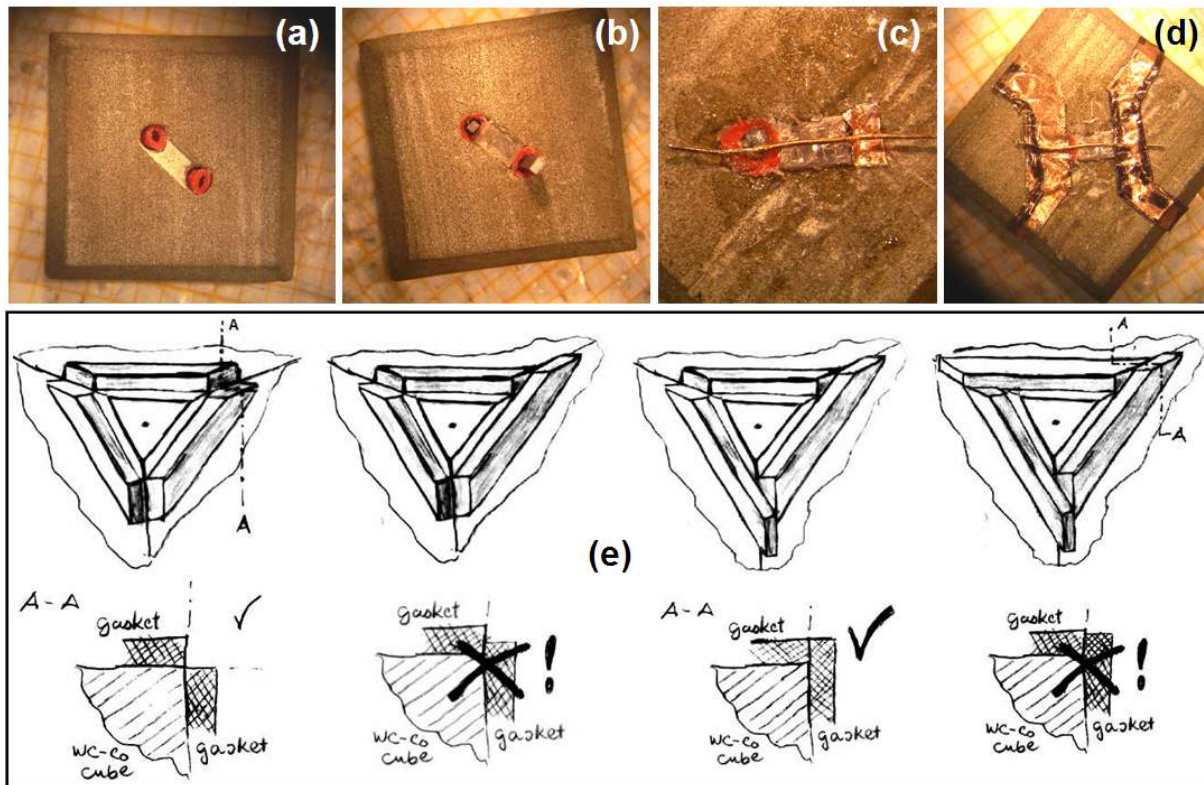
As outlined in Table 1, in order to give the students a chance to follow the resistance measurement *life* during compression, they are provided with a complete assembly (calibration octahedron and hard metal cubes, readily glued with gaskets, paper and epoxy sheets) pre-fabricated by either the previous group or the supervisor. The practical part of the course starts thus with the insertion of cubes and octahedron into the Walker module and consecutive wiring of the different calibrants to the measurement channels. However, before doing so, questions (i)–(iii) and in particular (iv) from Section 4, are briefly discussed. Moreover, the students have to explain why even four instead of the original three (c.f. question (iv)) calibrants can be measured with only 8 leads, if two materials are (initially) semiconductors.

In order to save time, the practical work is then partitioned between the students. While one or two are concerned with building up the circuitry according to the octahedron orientation of their choice (different octahedron orientations are possible that lead to different connexion schemes!) the others can be instructed how to program the pressure profile, use the data logging software or to prepare the next calibration octahedron and anvil assembly from the prefabricated parts given in Section 2.2. The latter is most effectively explained via a sequence of photographs or drawings (Fig. 2).

The micrographs (a)–(d) represent only a selection of the most prominent assembly steps. The true assembly sequence is more detailed, including e.g. cutting of the AgCl pressure medium or scraping-off the silk insulation from the manganin to make good electrical contact. The complete sequence is provided in the supplementary data.

⁴ This paper is in particular interesting when the number of students and experiments is large enough to allow for a similar systematic study as that performed by Walker. For each group, the experimental set-up could then be varied by one parameter, such as the presence or absence of a lubricant between certain sliding surfaces of the Walker module, gasket material or thickness and so on. . .

Figure 2: Examples for visual assembly instructions: (a) Pre-fabricated central part of the octahedron with inserted PTFE tubes, (b) insertion of the semiconductor pieces, (c) placing the manganin wire, (d) fixation of the wire and electrical leads with self-adhesive copper tape, (e) instruction for proper alignment of the pyrophyllite gaskets



After all measurement channels have been established and proved to work, the Walker module is closed and inserted into the press. Ideally, there is sufficient time left for the students to continue their assemblage of the next sample and ‘their’ set of carbide cubes, while keeping an eye on the resistance changes during compression. These are displayed as an R -versus-time graph on the PC-screen by the measurement program. The data recording is continued for the full compression-decompression cycle and the two data logs are then submitted to the students, e.g. via e-mail, on the following day.

6. Student’s report, data evaluation and selected results

After the experiment, the students are requested to provide a report, containing an introductory part and the evaluation of the measurement data. Following guiding questions were given for their report:

- Describe briefly the subject of this lab course
- Why must a multianvil press be calibrated? Why can the pressure at the sample not be just calculated by $p = \text{Force}/\text{Area}$?
- Which calibrants were used? Do all have the same or different modes of action?
- Make a sketch which...
 - ... displays the internal assembly of the pressure cell with the calibrants (preferably 3D view)
 - ... allocates the connexions between the measurement circuits inside the pressure cell and the different channels of the digital multimeter via the anvils

As mentioned in Section 2.1, the current setup of the experiment produces two data log files, called *R-Log* and *P-Log*. In order to extract the pressure-versus-ram force relation, the students are further instructed as follows:

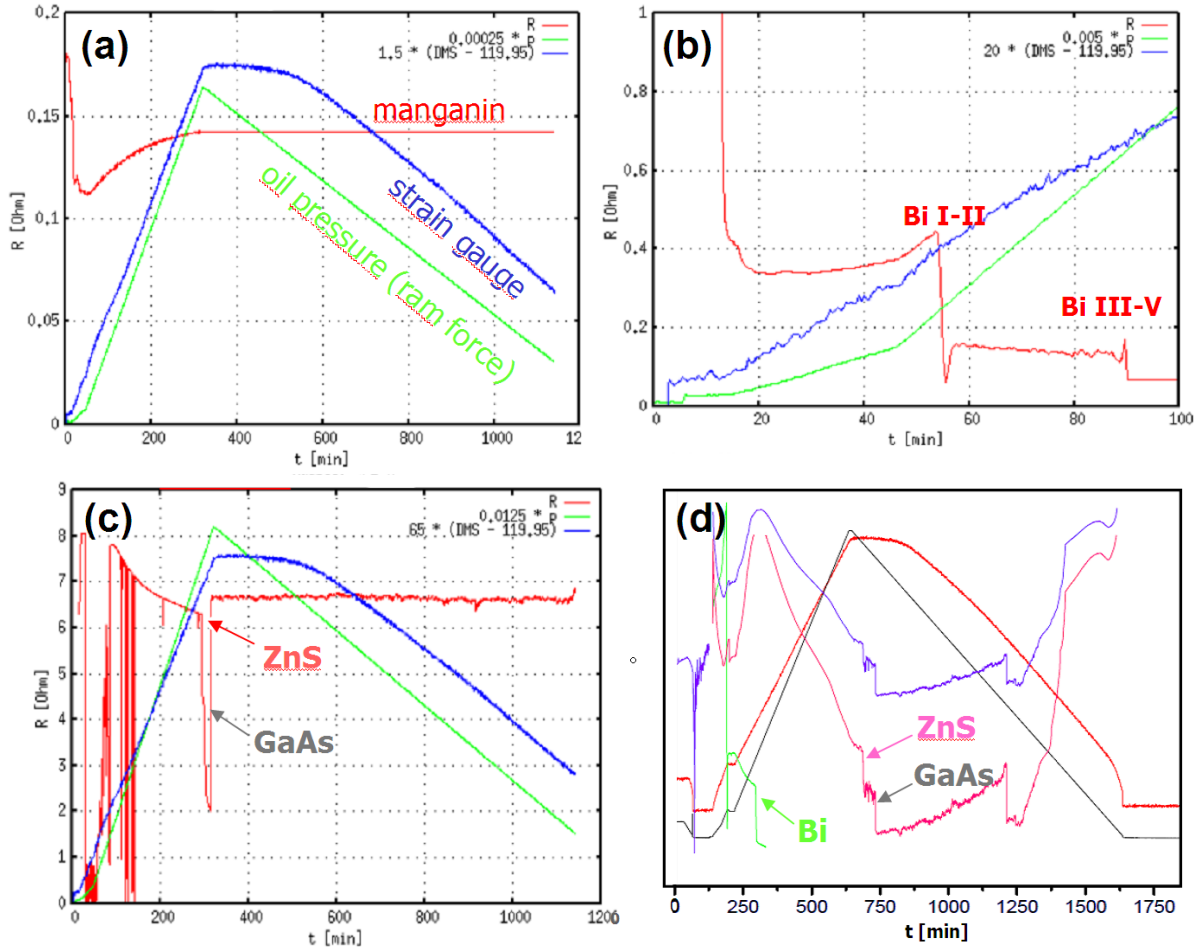
- unify the time scales of the R- and P-Log
- convert hydraulic oil pressure in the P-Log to ram force [tons]
- plot oil pressure, strain gauge and sample signals vs. the common time
- identify the fixed points, if necessary recall the corresponding literature (Sect. 4)
- plot R (manganin) and/or $\Delta R/R_0$, where $R_0 = R$ (manganin@Bi I-II) versus sample pressure p at each fixed point
- determine the pressure-dependence of the manganin resistance and the manganin gauge factor⁵ by linear interpolation
 - fitting with Bi I-II and III-V transitions only
 - fitting Bi and/or ZnS and/or GaAs transitions
 - is there an outlier? Which semiconductor transition is more trustworthy with respect to purity?
- finally plot the sample-pressure versus ram-force relation, including the fixed points, as well as continuous pressure curves based on established pressure-dependence of the manganin signal
- discuss the results with respect to published multianvil calibration data (e.g. the interlaboratory comparisons presented at <http://multianvil.asu.edu>)

Alltogether, four calibration runs were performed. The main difference between these runs were the gasket thickness and material (run No. 1: 2.0 mm pyrophyllite, run No. 2: 2.2 mm pyrophyllite, run No. 3 and 4: 2.0 mm organic filler + epoxy resin). The measurements in run No. 3 lost electrical contact due to very strong gasket extrusion and could not be evaluated. In Figure 3 the results of the above evaluation procedure are shown for run No. 2 and 4 – as taken from the reports of student group 3 and 4 (see Acknowledgements). Panel (a)-(c) is from a run (No. 2) where all measurements worked. Manganin shows a continuous increase in resistance upon compression, but interestingly remains constant upon decompression. The strain gauge shows typical hysteresis effects during the decompression, as already noted by Walker et al. [5]. The Bi transitions are well resolved and the first one already starts shortly after the pre-compression phase (smaller slopes in the oil pressure and strain gauge record) at a ram force of about 50 tonnes. The semiconductor-to-metal transitions of ZnS and GaAs appear as consecutive kinks in one single curve as they are effectively measured in parallel.

This behaviour can be seen to be reproducible in Panel (d) of Fig. 3. However, in this case the transitions appear *after* the same maximum ram force was reached. This indicates a lower efficiency and some creep behaviour of the newly tested gasket material. Possibly due to the fact that the epoxy resin has less friction with the tungsten carbide anvils than the traditional pyrophyllite. If regardless of this fact the fixed points of run No. 4 are plotted versus ram force (Fig. 4 (b)), this leads to the somewhat strange situation that the ZnS transition appears to occur at higher load than the one GaAs. However, as can also be seen from Fig. 4, the pyrophyllite-gasketed experiments were well-behaved, showing a somewhat higher efficiency for the thinner gaskets at high pressures. In panel (a) of Figure 4, an excellent correlation between manganin resistance of experiment No. 1 and the literature values for the fixed point pressures of the Bi I-II, III-V and GaAs transformations can be seen. The coefficients of the two linear fits differ only marginally. For ZnS the *true* transformation pressure must be higher than the literature value of 15.5 GPa (Onodera et al. [10]) in all cases. However, literature values for the ZnS transition pressure tend to scatter largely, ranging from 13.8 to 18.5 GPa (see [10] and references therein), depending on the stress-state inside the pressure medium. In the present

⁵ i.e. the slope of relative resistance $\Delta R/R_0$ versus pressure

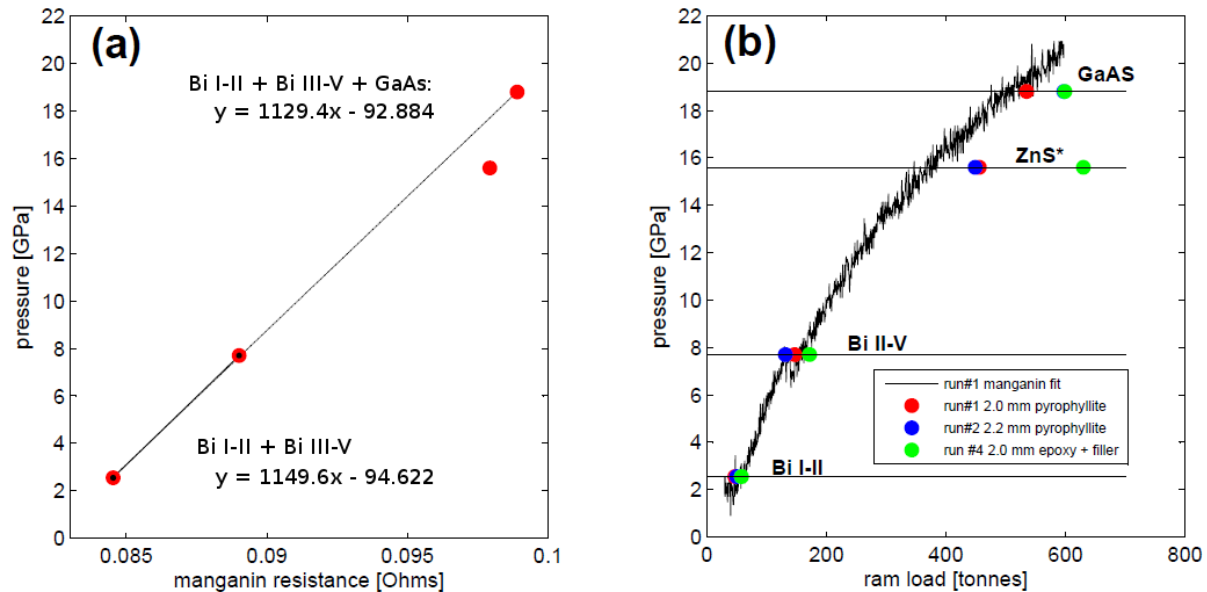
Figure 3: (a)-(c) Plots from the data evaluation of high-pressure run No. 2 (report of student Group 4), (d) Plots from the data evaluation of high-pressure run No. 4 (report of student Group 3)



case, also the fact that the employed ZnS was a natural ore with higher impurity level could have played a role. It was excluded for the fitting the manganin resistance data.

Finally in panel (b) of Figure 4, the great advantage offered by the manganin resistance measurement can be seen. It produces a true continuous trace in the ram-force-versus-sample-pressure graph up to pressures above the last fixed point transition. Fitting this trace instead of the fixed points should thus lead to a more accurate calibration curve. In the case of run No. 1, fitting of a quadratic polynomial gave the following result: $p = -3 \cdot 10^{-5} \cdot F^2 + 0.0518 \cdot F + 0.4477$ with p = sample pressure in GPa and F is ram force in metric tons.

Figure 4: (a) Correlation between manganin resistance and fixed points of run No. 1, (corresponding pressure data taken from literature) (b) ram-force-versus-sample-pressure relationship ('multianvil calibration curve') of the 3 successful experiments, the black trace is the manganin resistance signal of run No. 1, converted into a pressure scale, using the linear fit for the Bi transitions (lower equation in panel (a)), the curve for the Bi + GaAs fit deviates only marginally



7. Résumé of the 1st course held in Jan./Feb. 2009

A multiple-probe method for determining the entire force-vs.-pressure relation of multianvil-devices at room temperature in a single run has been developed. It was demonstrated, that this method is feasible as an interesting, interdisciplinary and multi-tasking experiment for students with either scientific or engineering background. Despite its complexity, the lab course can basically be prepared and supervised at any time by a single assistant and on a time scale that is essentially comfortable for both, teacher and students. Nevertheless, proper preparation and execution of the experiment requires a high degree of coordination and partitioning of work (working in parallel) among the students. It hence was experienced that several groups required more time than originally set out in the schedule (typically 5 h, from 8:00 am till 1:00 pm). Interestingly, the somewhat tedious assembly of the calibration octahedron turned out to be a minor problem, for there was always at least one student with sufficient natural skill to do the job without prior training. This was most likely due to the fact that there was a clear one-after-the-other instruction (cf. Fig. 2 (a)-(d) and supplementary information). Help was in most cases only needed for cementing/glueing of the three finished octahedron parts. Instead, two other parts of the practical work turned out to be the main causes for delay: (i) due to the attachment of gaskets (recall Fig. 2 (e)), differently-sized epoxy sheets, and electrical leads (c.f. Fig. 1(b)) to the second stage anvils, these previously symmetric tungsten carbide cubes become essentially chiral—even unique with respect to their position within the walker-module and the respective connection wires, thus making their assembly into an interesting three-dimensional puzzle, not unlike Rubik's 'magic cube'. A simplified method that comes along without adhesive copper tape to be attached to the cubes is currently being devised. It might also help to eliminate the loss of electrical contacts, which have been occurred during some of the high pressure runs resulting in incomplete data records. (ii) closely connected to the above-mentioned problem is the final orientation of the calibrant octahedron within its cavity and the consecutive wiring of the different calibrants to the measurement channels (c.f. Fig. 1(b)). Other than the above-noted task however, this one is especially critical. First, since an error here could easily spoil the entire measurement, the connexion scheme has to be carefully cross-checked by both, the

students themselves and the assistant. Second, this task *must* be finished before the compression can start. Hence, a delay here limits the time available to the students to follow the resistance measurement actually *in situ*. In the worst case, they will only obtain the resistance record files afterwards. Nevertheless, as this task is meant to be an essential part of the training, it cannot be further simplified. It may be thus better to allocate in the order of 6 h for each group.

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