HELMHOLTZ ASSOCIATION

GFZ POTSDAM

International Workshop on Synchrotron High-Pressure Mineral Physics and Material Science
Advanced Photon Source
Argon National Laboratory
Argonne, Illinois USA,
December 6-7, 2007

Ultrasonic Measurements in Multi-Stage Multi-Anvil Devices

Hans J. Mueller, Frank R. Schilling, Michael Wehber, Christian Lathe

Outline

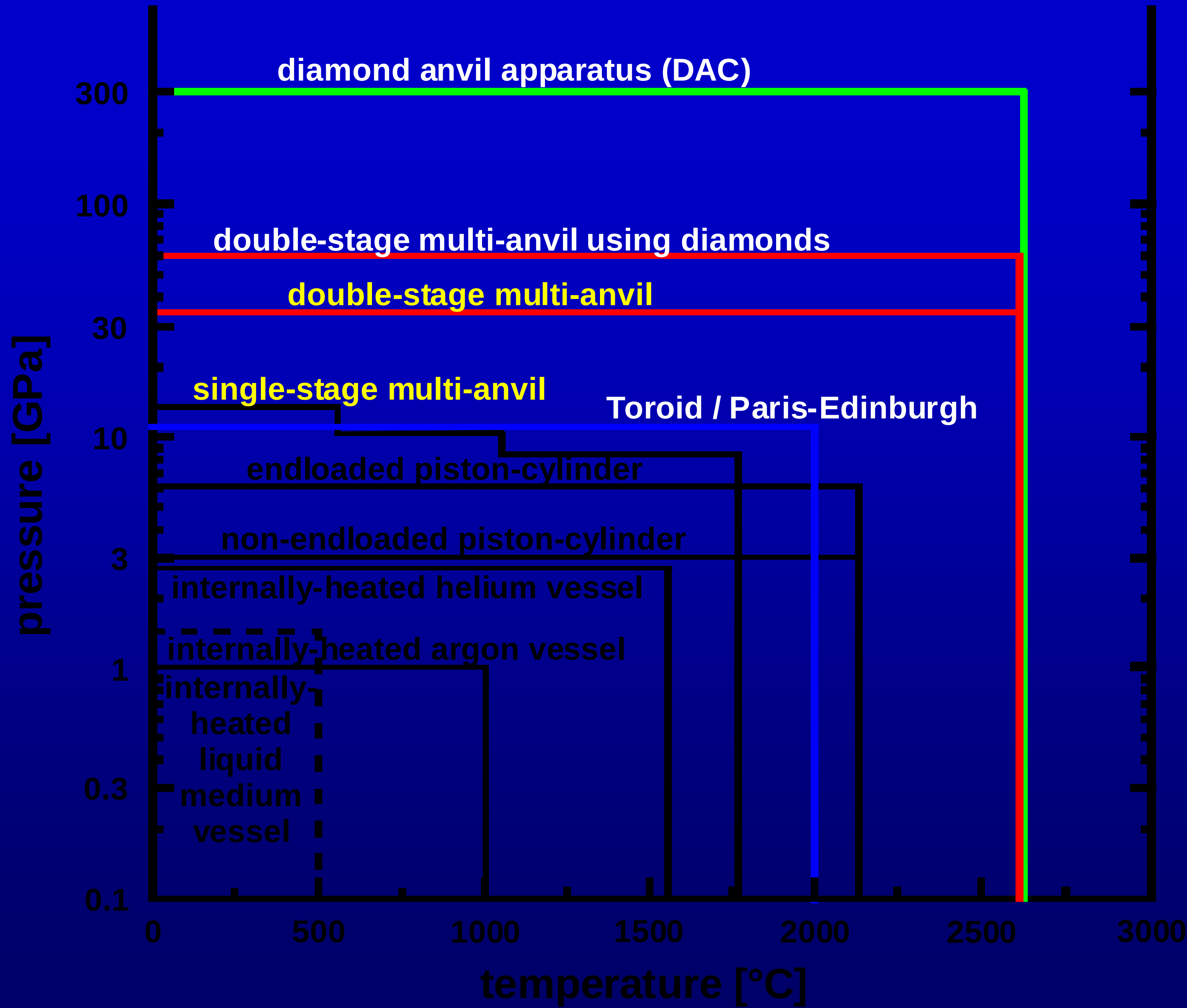
High-pressure techniques

Additional opposed anvil stage in LVPs

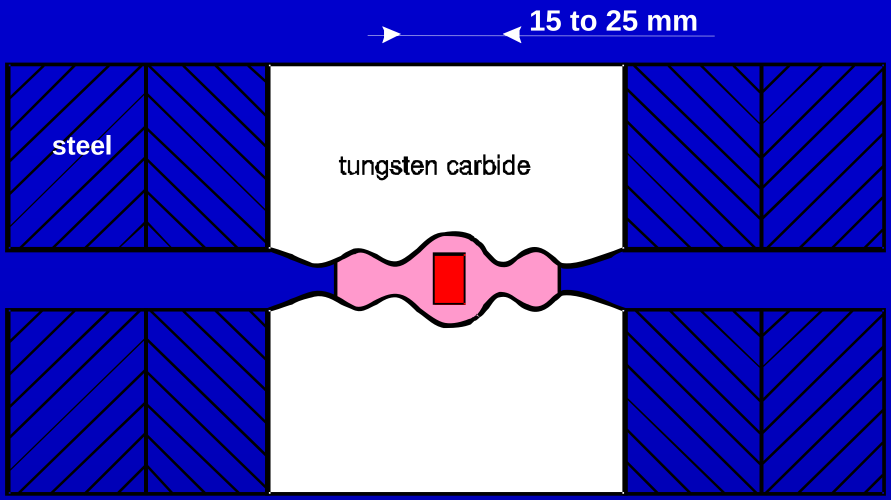
Multi-stage experiments at DESY-HASYLAB – MAX80 & MAX200x

Ultrasonic multi-stage experiments

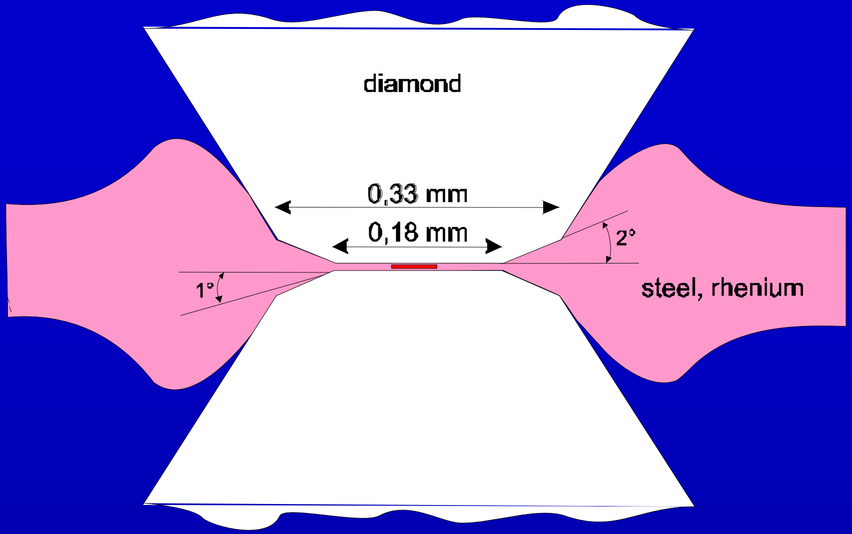
Standard-free pressure measurement



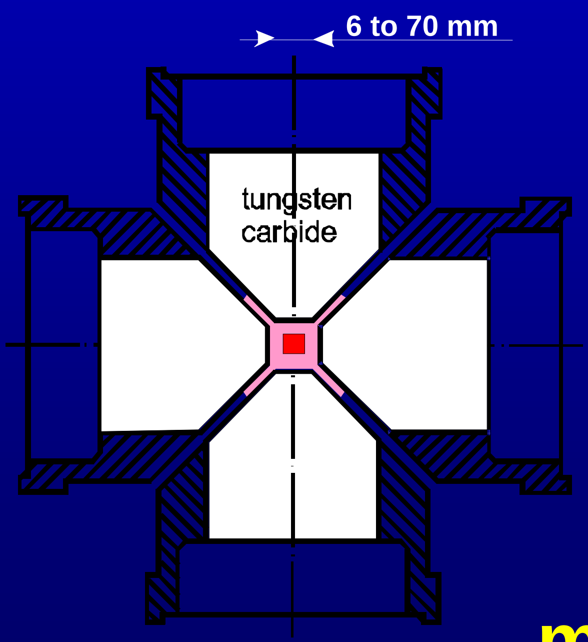
Hans-Joachim.Mueller@gfz-potsdam.de



toroid



DAC



multi-anvil

Simulation of mantle conditions with synchrotron radiation access

Hans-Joachim.Mueller@gfz-potsdam.de

2nd stage opposed anvils

X-ray diffraction under ultrahigh pressure generated with sintered diamond anvils

W. Utsumi, N. Toyama, S. Endo, and F. E. Fujita
High Pressure Research Laboratory, Faculty of Engineering Science, Osaka University, Toyonaka, Osaka 560, Japan

O. Shimomura
National Institute for Research in Inorganic Materials, Sakura-mura, Niihari-gun, Ibaraki 305, Japan

(Received 3 March 1986; accepted for publication 13 June 1986)

High pressures generated with sintered diamond as the second stage anvils in the "6-2 type" double-stage apparatus have been calibrated by x-ray diffraction with synchrotron radiation. Sintered diamond has permitted a fair amount of x-ray transparency although the chemical analysis shows the existence of 14-wt. % Co binder. Anvils with a top face of 0.7 mm diam performed the generation of 60 GPa, which was confirmed on the lattice parameters of *B* 1 and *B* 2 phases of NaCl. No appreciable plastic deformation of the used anvils has certified the higher pressure generation.

I. INTRODUCTION

Sintered diamond (abbreviated as SD, hereafter) is a new and promising anvil material to generate ultrahigh pressure. SD is expected to be very tough since the cleavage, which is characteristic to single-crystal diamond, is not propagated through but arrested by the diamond particles constituting SD. In addition, the preparation of SD aggregates much larger than the usually obtainable single crystals is possible and less expensive. The high compression strength or hardness is already tolerable for producing pressures up to about 60 GPa and will be further increased by improving the technique of preparation and application. Bundy[1,2] successfully used the SD anvils in a Drickamer-type high-pressure cell and reported the generation of 50 GPa based on the linear extrapolation of the pressure-applied load relation. We have also carried out the sintering of diamond particles and developed a multistage system using the SD as the final stage anvils, by which an electrical resistance measurement and a mineral synthesis were made under high pressures.[3,4]

At present, there is no established pressure-fixed point above 25 GPa, so that other calibration methods must be applied to exactly evaluate the higher pressures generated with SD anvils. The ruby fluorescent method conventionally used in the single-crystal diamond anvil technique is not applicable because SD is opaque due to the presence of binder material used for sintering diamond particles. A similar problem has also been pointed out for the use of the x-ray diffraction method. We report, in the present paper, a successful attempt of an x-ray diffraction technique to measure the high pressures generated with SD anvils by utilizing a strong source of synchrotron radiation (SR).

II. PREPARATION OF SINTERED DIAMOND ANVIL

SD anvils used in the present study were produced in the High Pressure Research Laboratory (HPRL) of Osaka University. The sintering process has been briefly described elsewhere.[4] Diamond powder of two different particle sizes of 60 μm and 12–25 μm mixed with the ratio of 7:3 is packed in the hole of a WC–Co ring. At high temperature and pressure in the diamond stable region, molten cobalt extrudes from the ring, penetrates into the gap between diamond particles, and eventually cements them closely by the liquid phase sintering process as shown in an electron micrograph of Fig. 1, in which the white color corresponds to Co element as a result of EPMA observation. Formation of direct diamond–diamond bonding is seen at some places although the number is limited. The particle size of diamond and the original cobalt

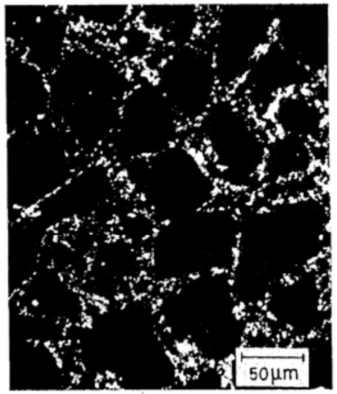


FIG. 1. Secondary electron micrograph of sintered diamond used in the present study. White parts are the areas at which Co element was detected by EPMA.

Hans-Joachim.Mueller@gfz-potsdam.de

Utsumi et al., 1996

2nd stage opposed anvils

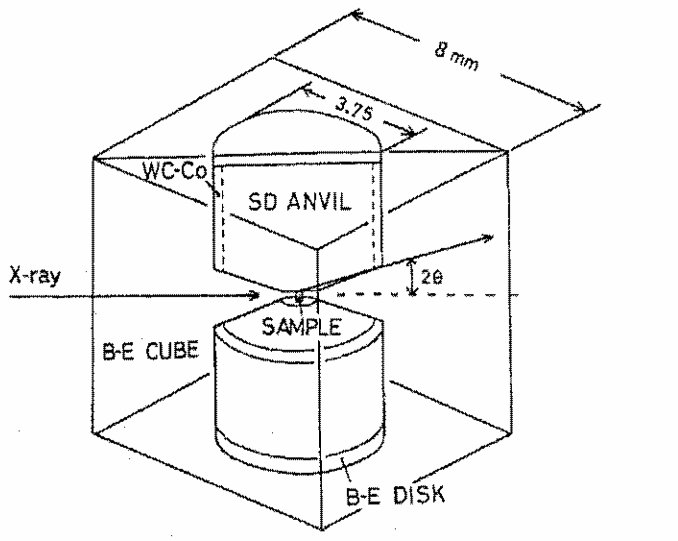


FIG. 3. Semi-cross section of the cell for "6-2 type" double stage system driven with the cubic press "MAX80." A pair of SD anvils with thin WC-Co outer ring sheath are embedded in a boron-epoxy (B-E) cube.

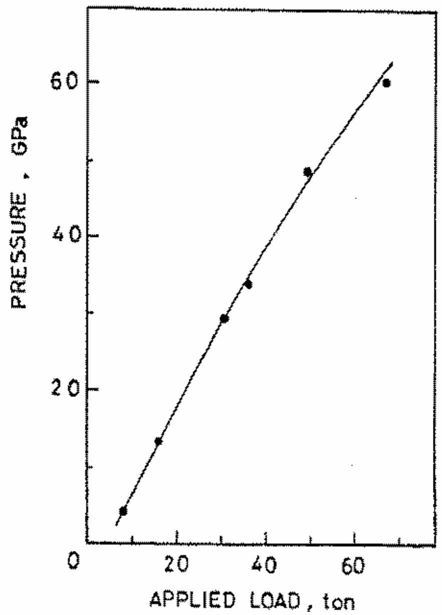
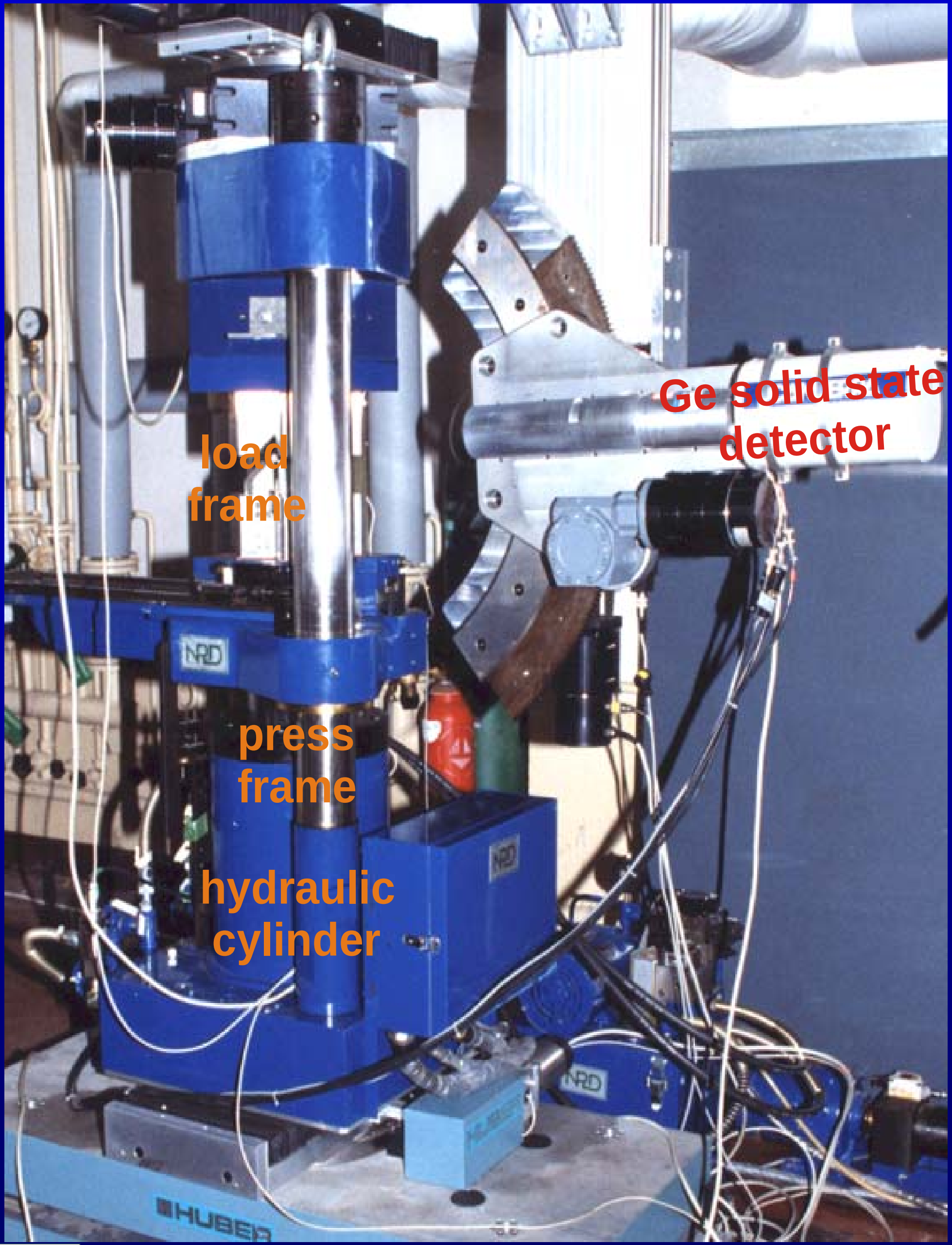


FIG. 5. Relation between pressure and applied load by MAX80 obtained from the patterns in Fig. 4.

Hans-Joachim.Mueller@gfz-potsdam.de

Utsumi et al., 1996

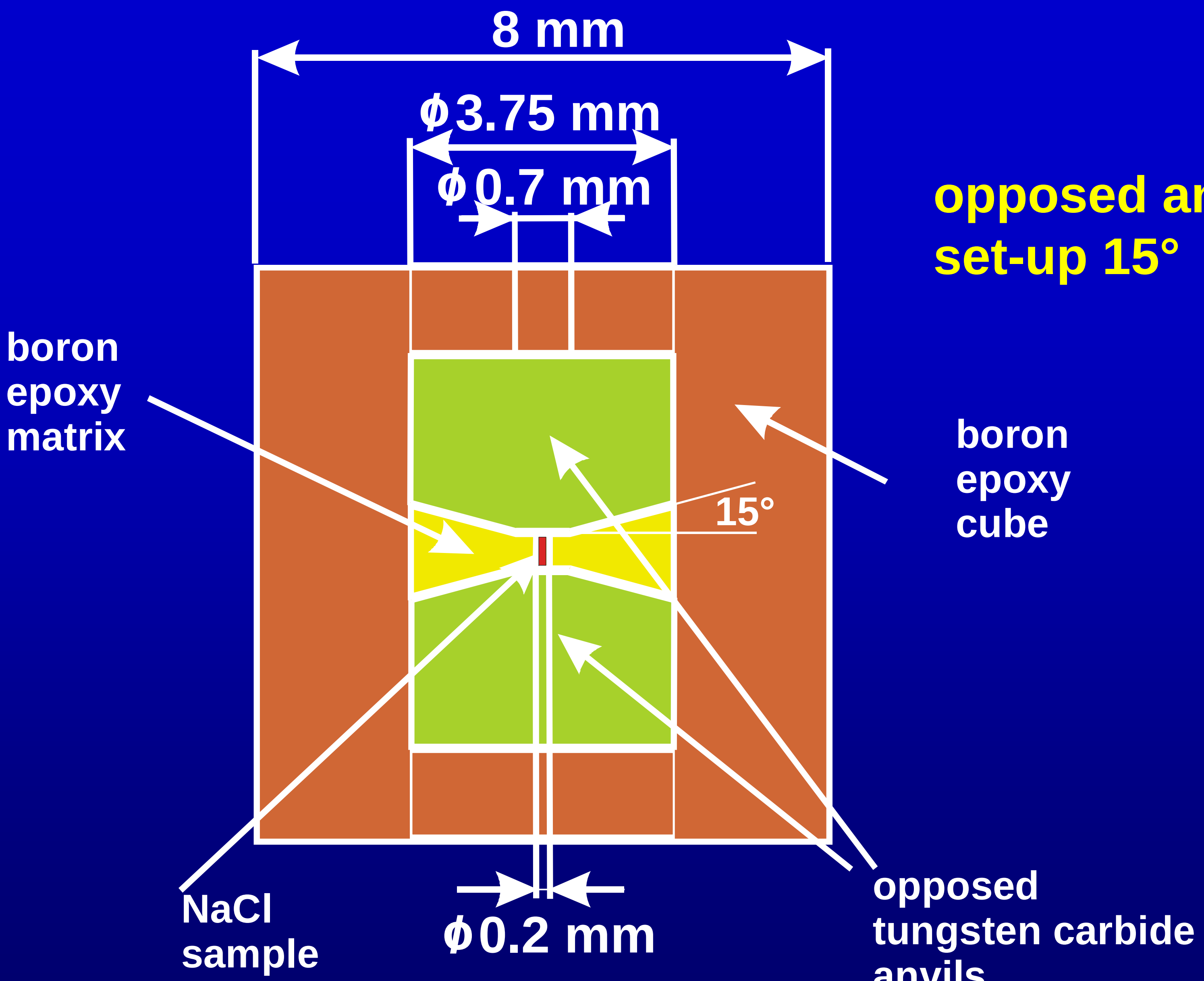


MAX80 - single-stage multi-anvil apparatus at DESY-HASYLAB

**maximum load:
250 tons**

GFZ POTSDAM
Hans-Joachim.Mueller@gfz-potsdam.de

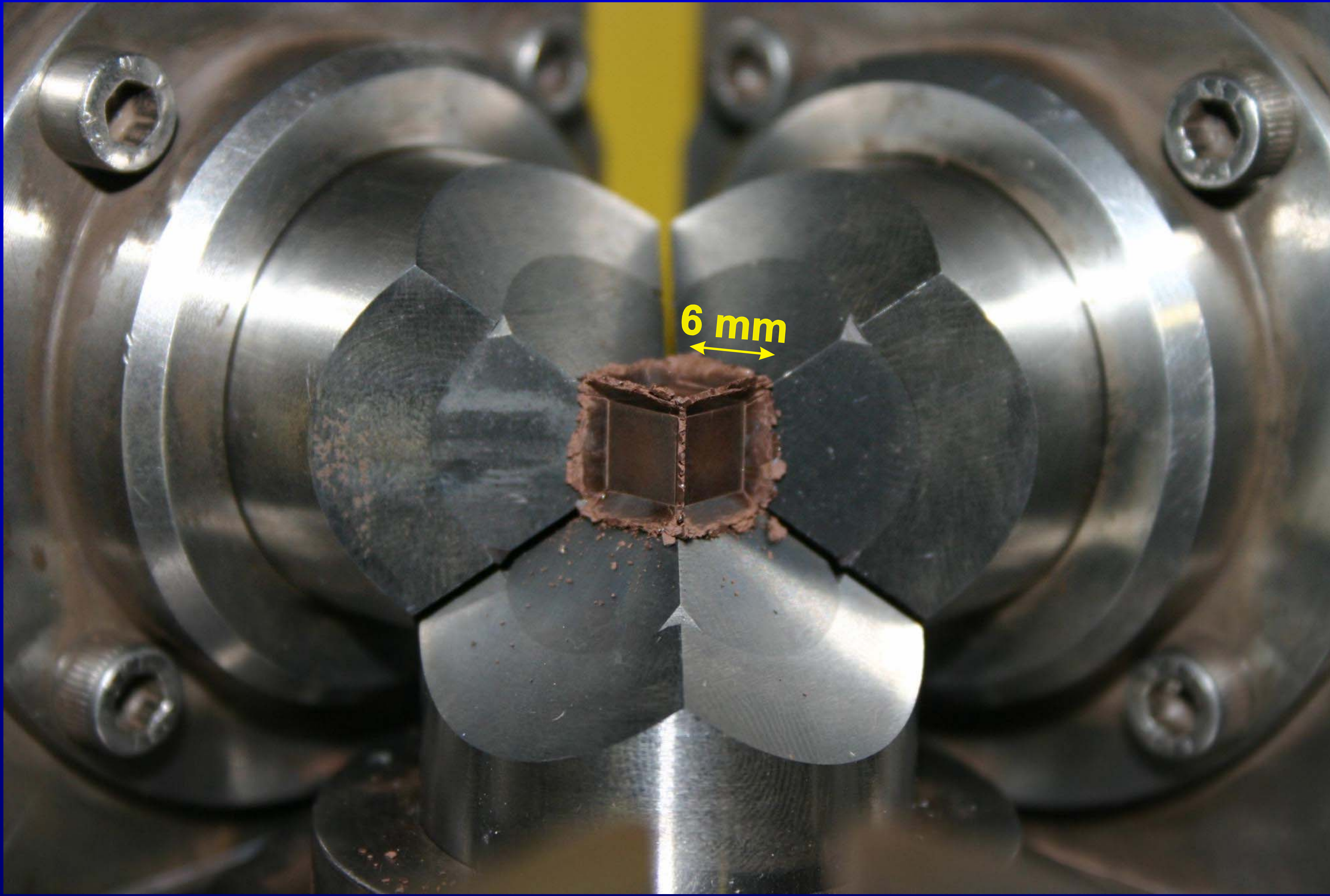
opposed anvil set-up 15°



based on Utsumi et al., 1996

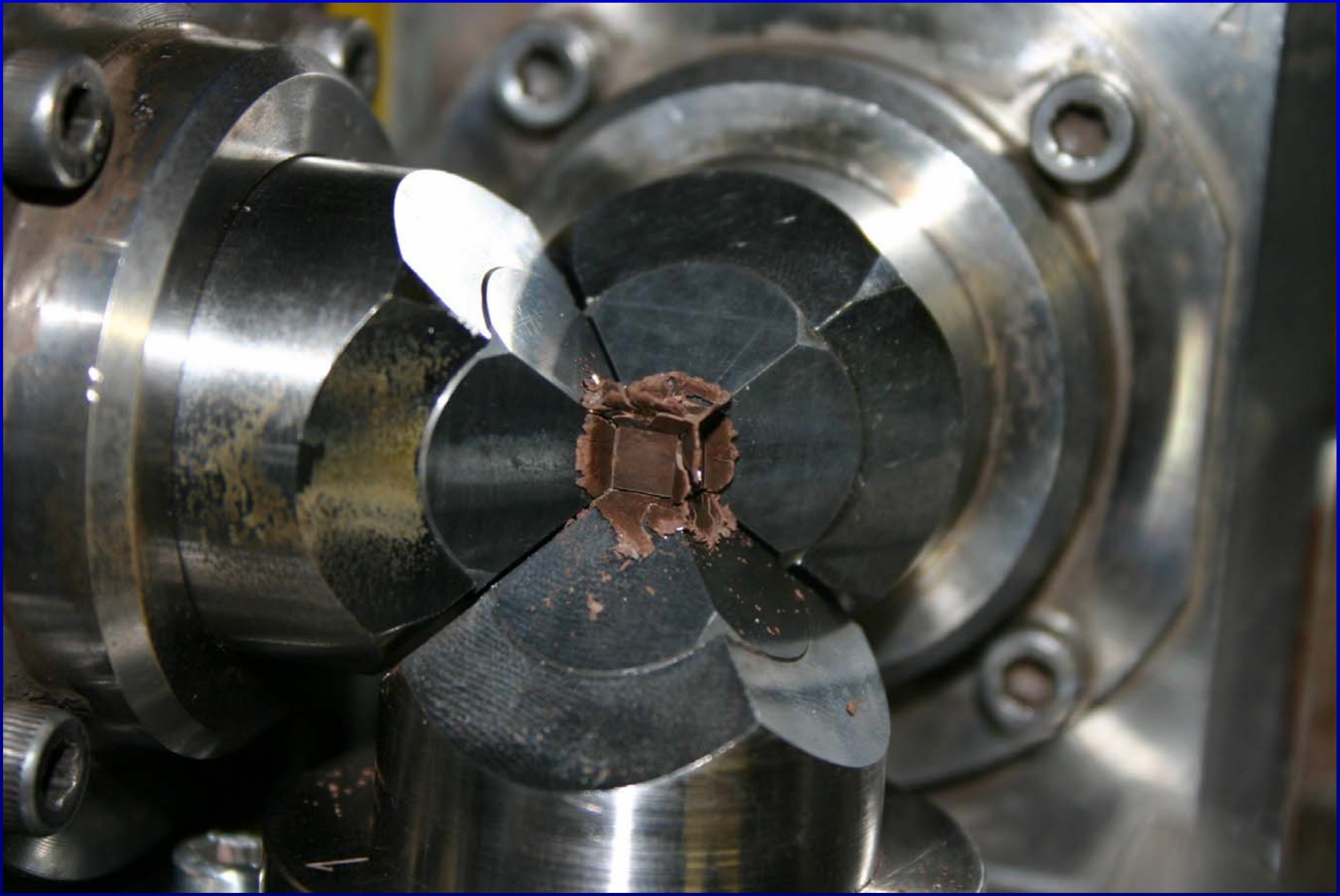
Mueller et al., 2006a

Hans-Joachim.Mueller@gfz-potsdam.de



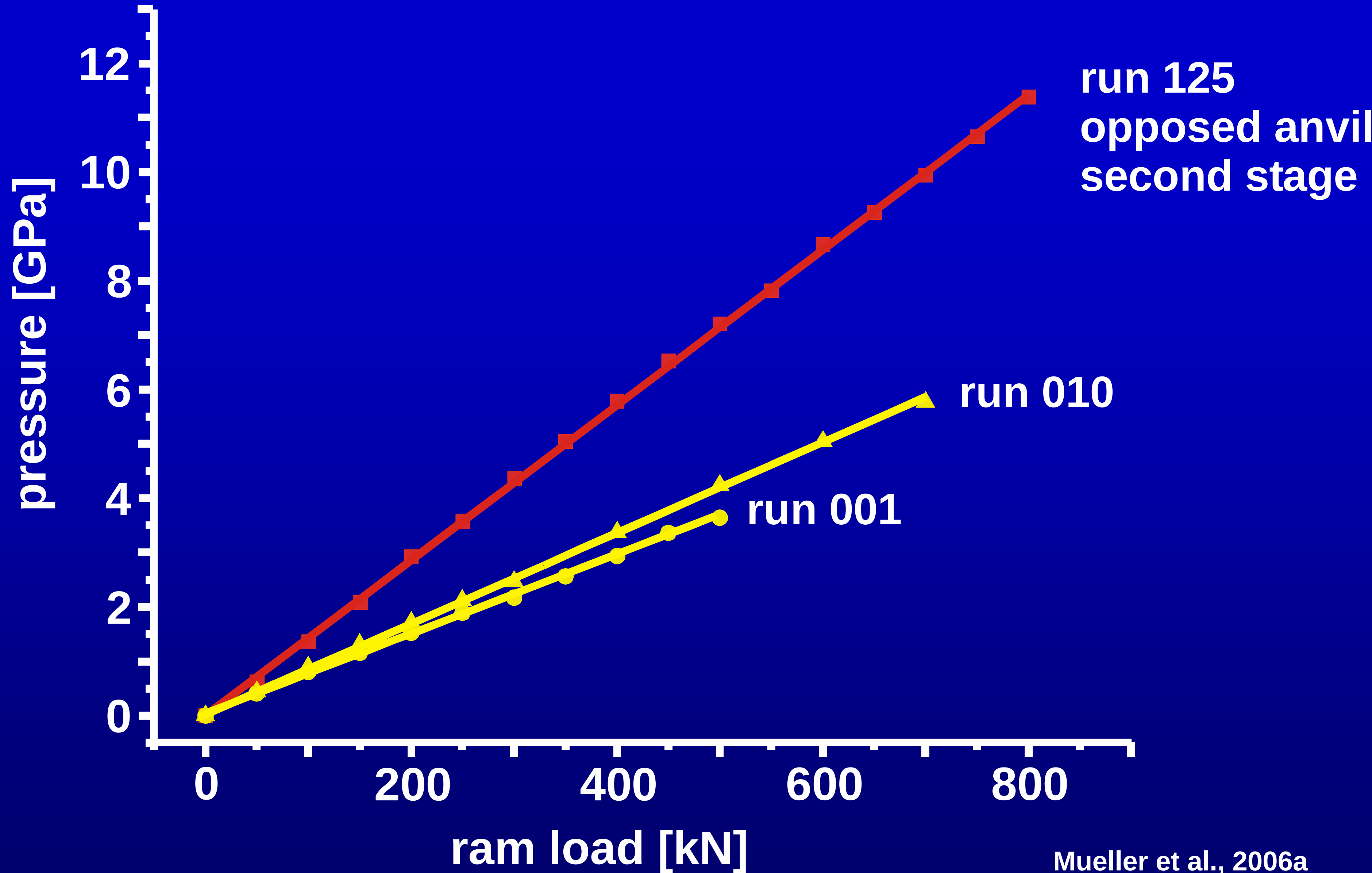
MAX80 – 2nd stage opposed anvil set-up after run

Hans-Joachim.Mueller@gfz-potsdam.de



MAX80 – normal single stage set-up after run

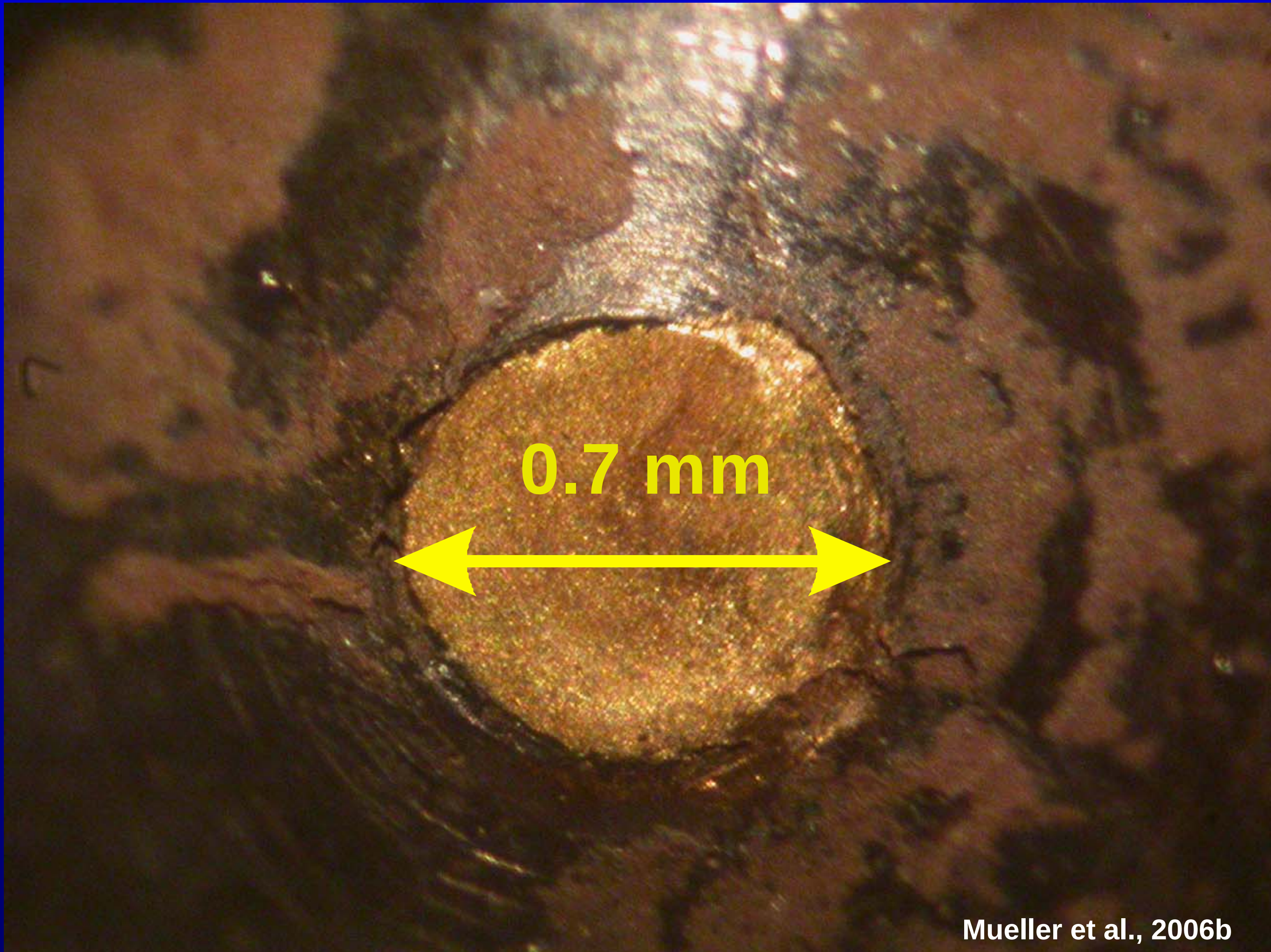
Hans-Joachim.Mueller@gfz-potsdam.de



Mueller et al., 2006a

pressure doubling by opposed anvils

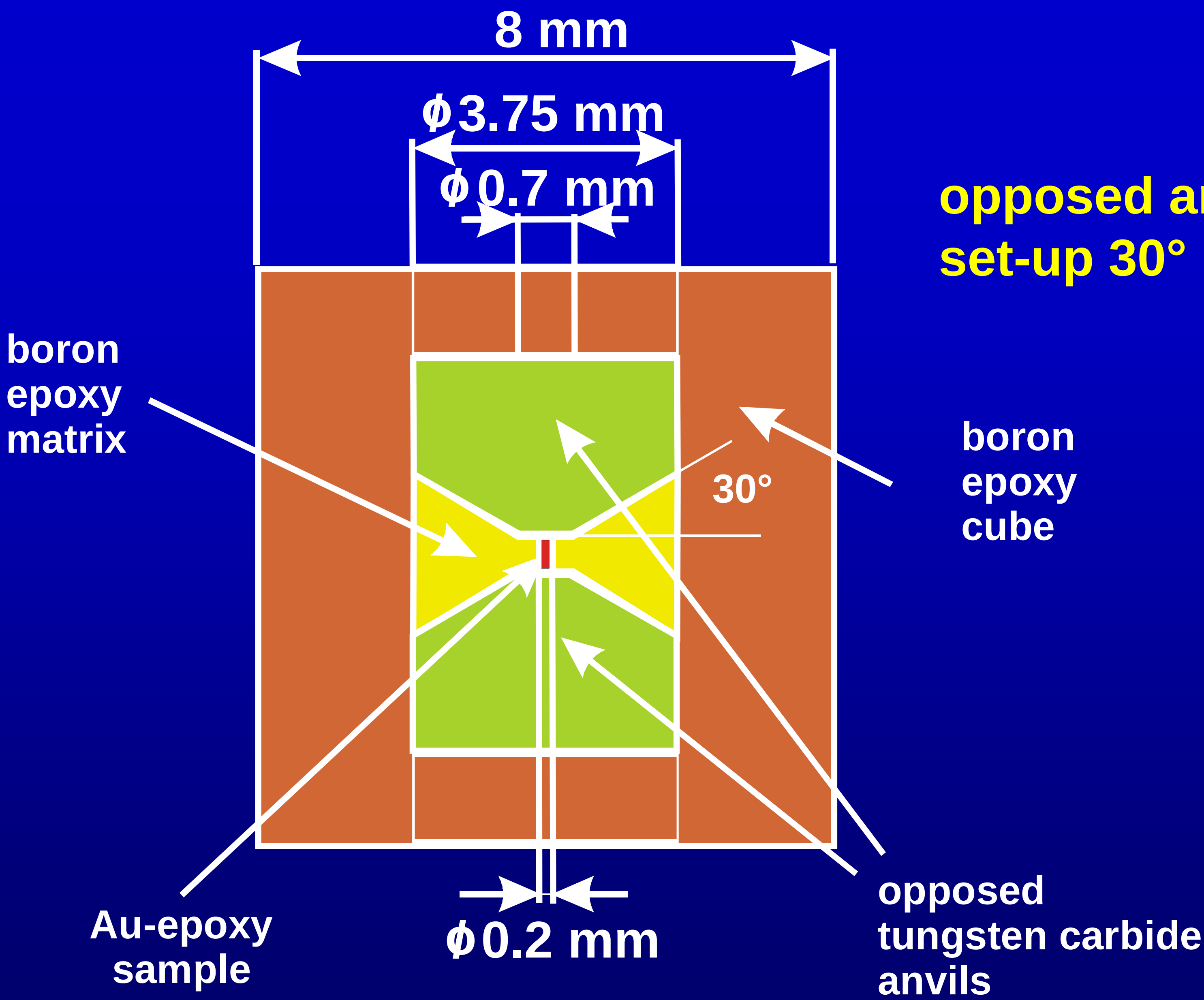
GFZ POTSDAM
Hans-Joachim.Mueller@gfz-potsdam.de



front face of an opposed anvil after the hp-run

Hans-Joachim.Mueller@gfz-potsdam.de

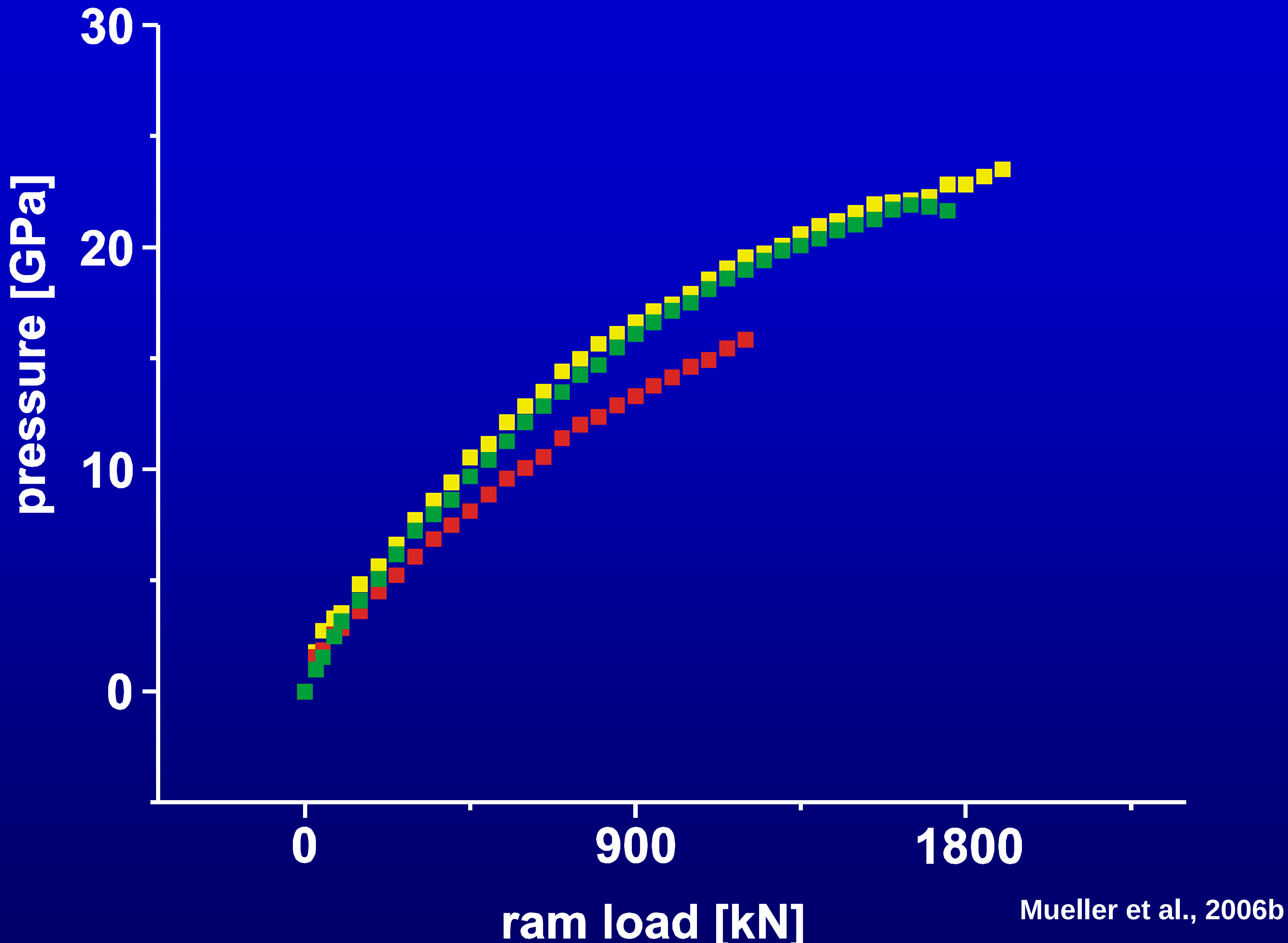
opposed anvil set-up 30°



based on Utsumi et al., 1996

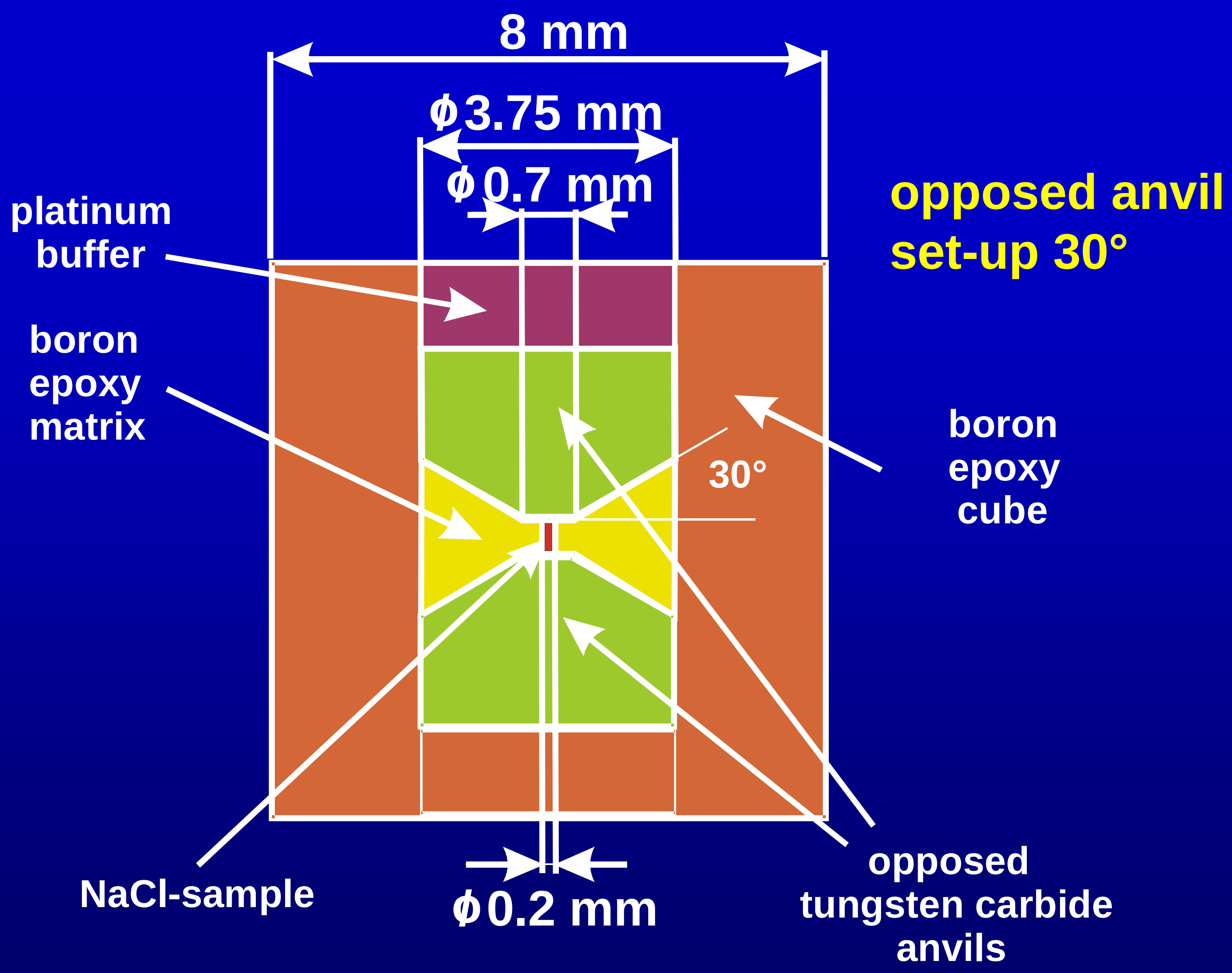
Mueller et al., 2006b

Hans-Joachim.Mueller@gfz-potsdam.de



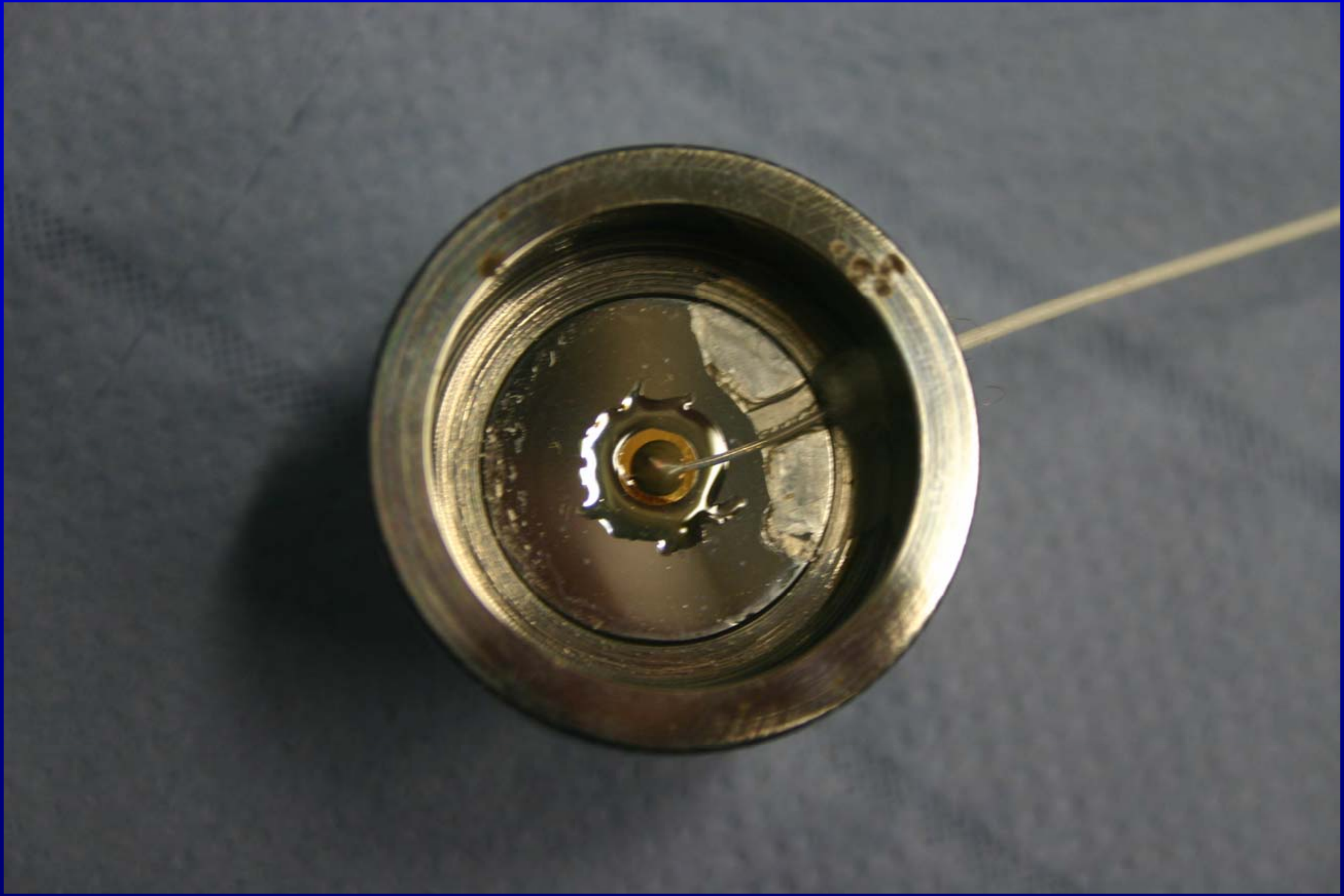
4-fold pressure increase by using 30° opposed anvils

Hans-Joachim.Mueller@gfz-potsdam.de



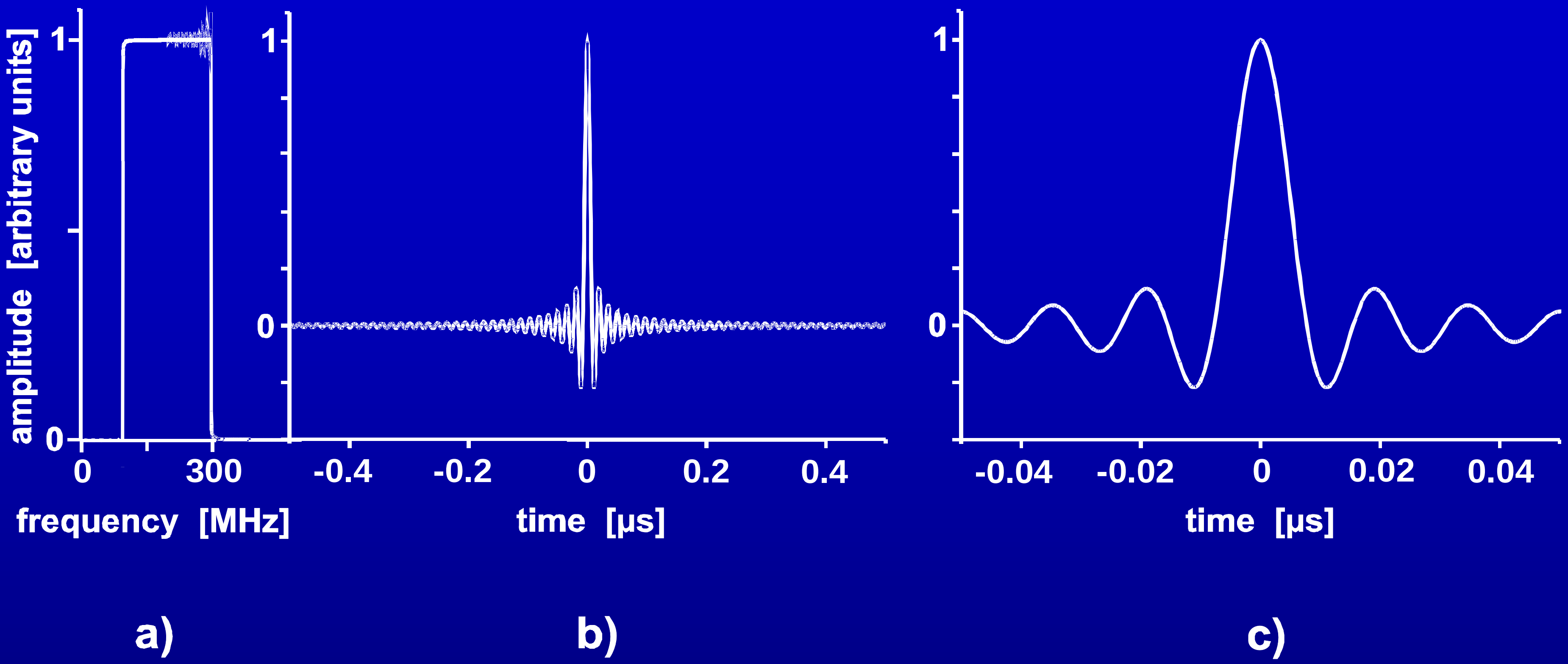
opposed anvils ultrasonic set-up

Hans-Joachim.Mueller@gfz-potsdam.de



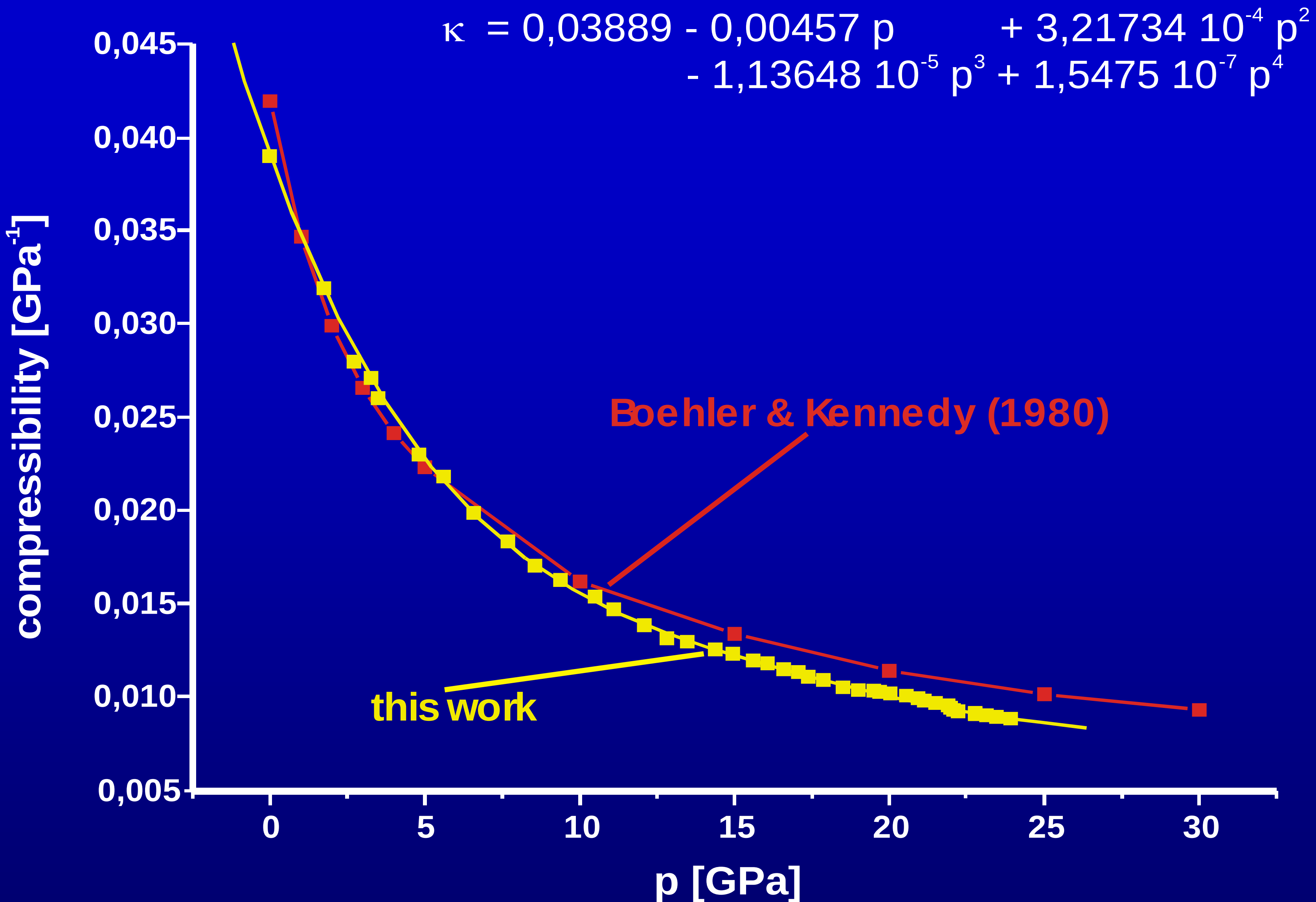
Triple-mode transducer at the polished rear side of MAX80's top anvil

Hans-Joachim.Mueller@gfz-potsdam.de



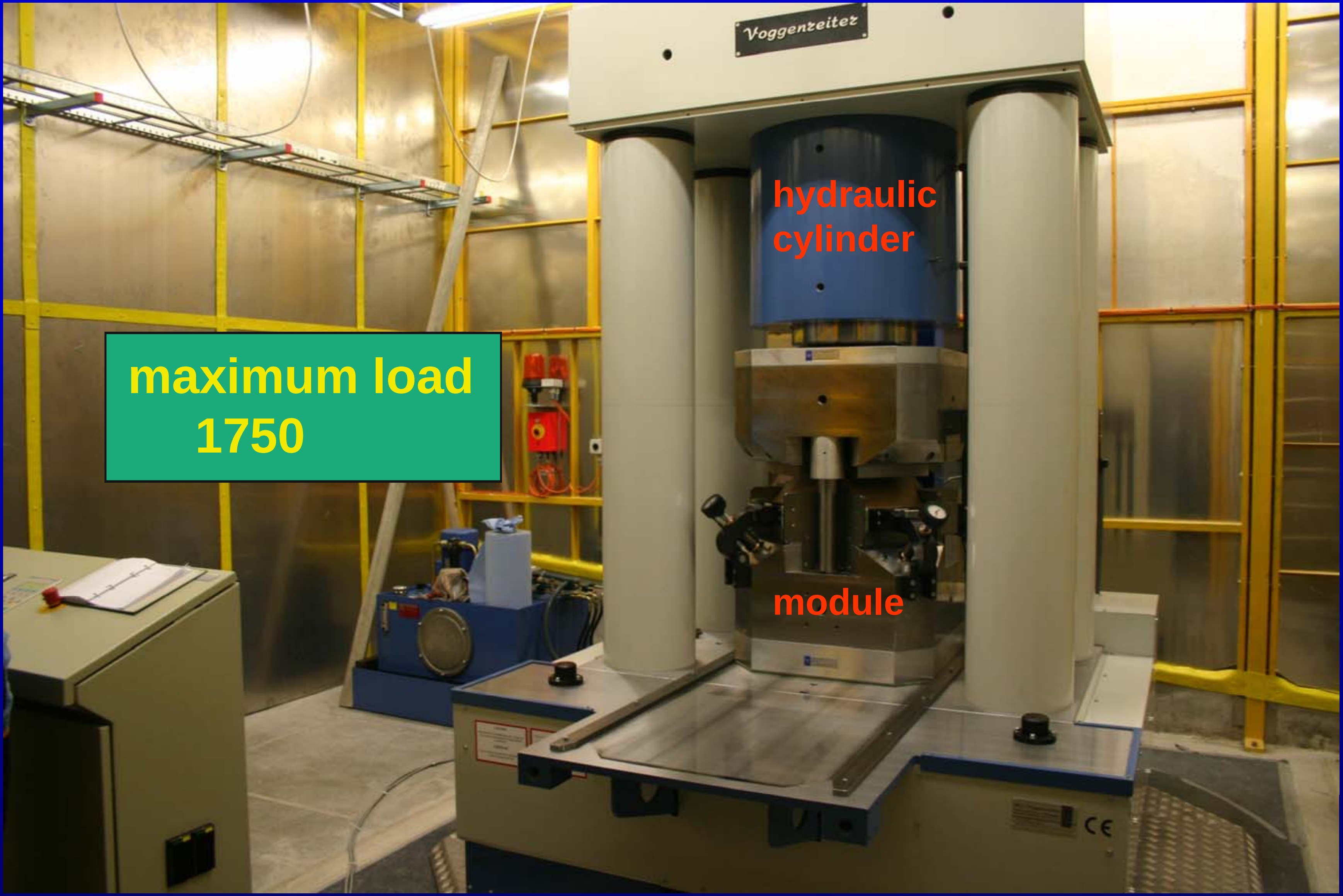
excitation function for opposed anvils ultrasonic set-up

GFZ POTSDAM
Hans-Joachim.Mueller@gfz-potsdam.de



compressibility of NaCl measured by ultrasonic interferometry

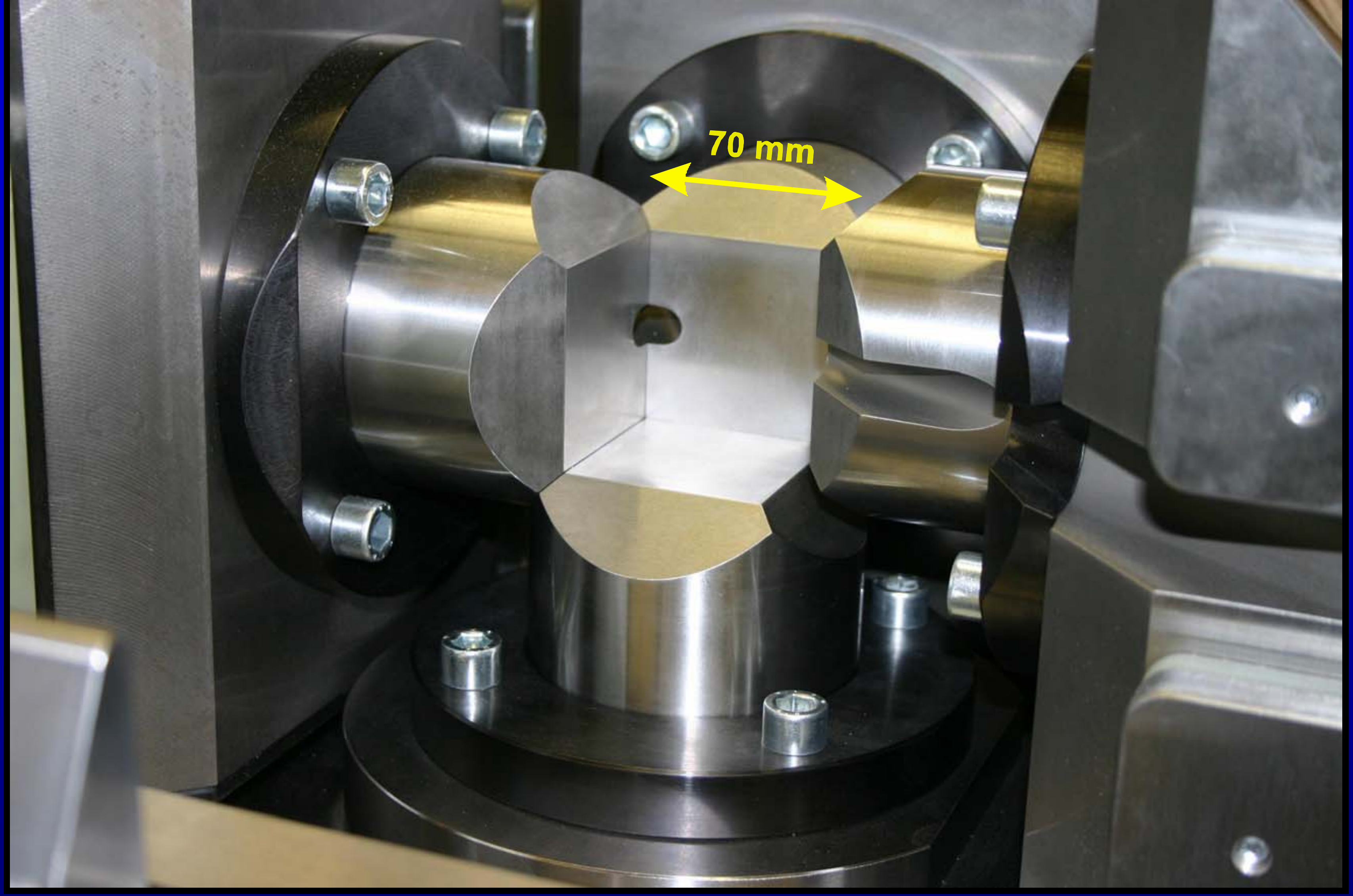
Hans-Joachim.Mueller@gfz-potsdam.de



MAX200x – double-stage DIA

GFZ POTSDAM

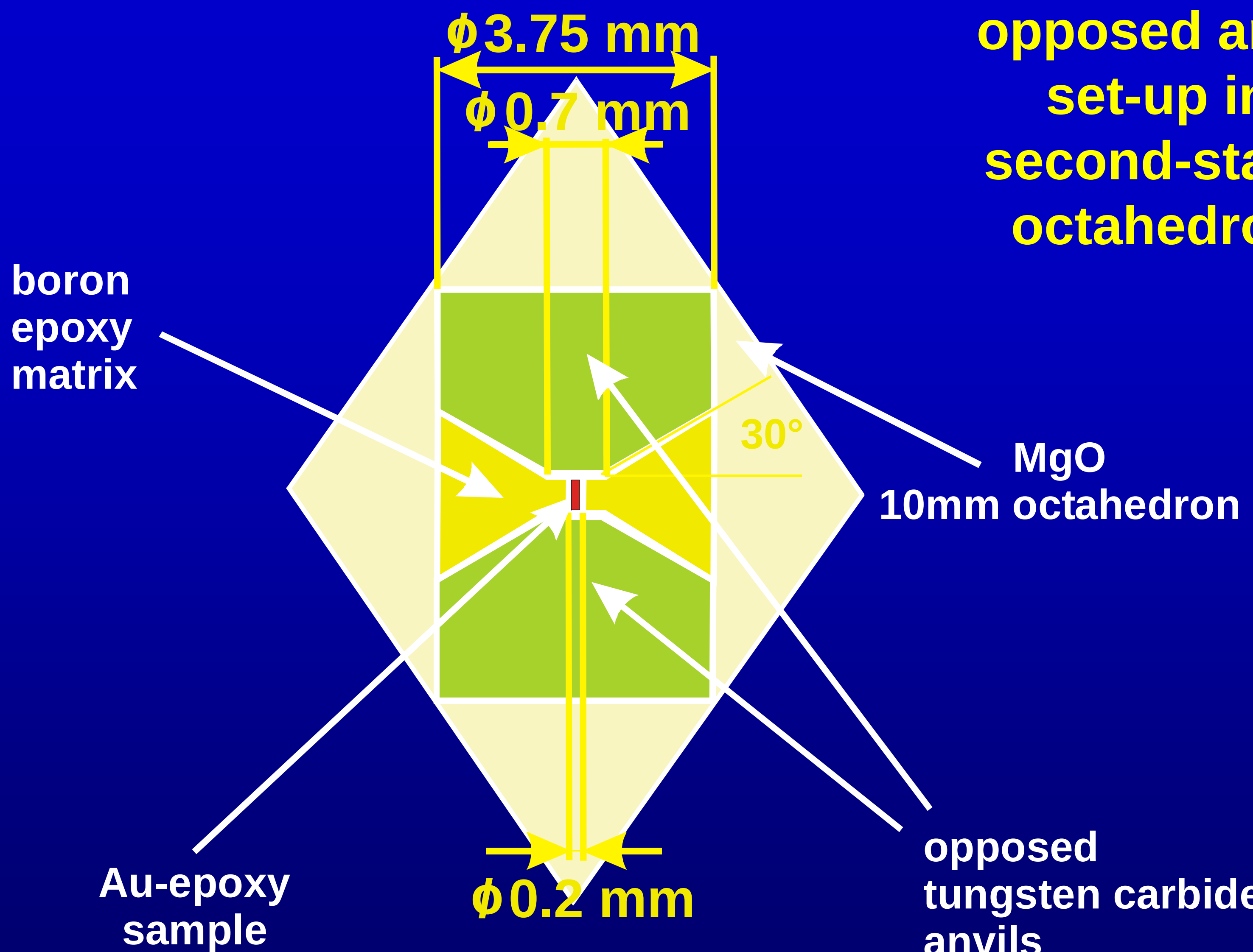
Hans-Joachim.Mueller@gfz-potsdam.de



MAX200x – DIA module

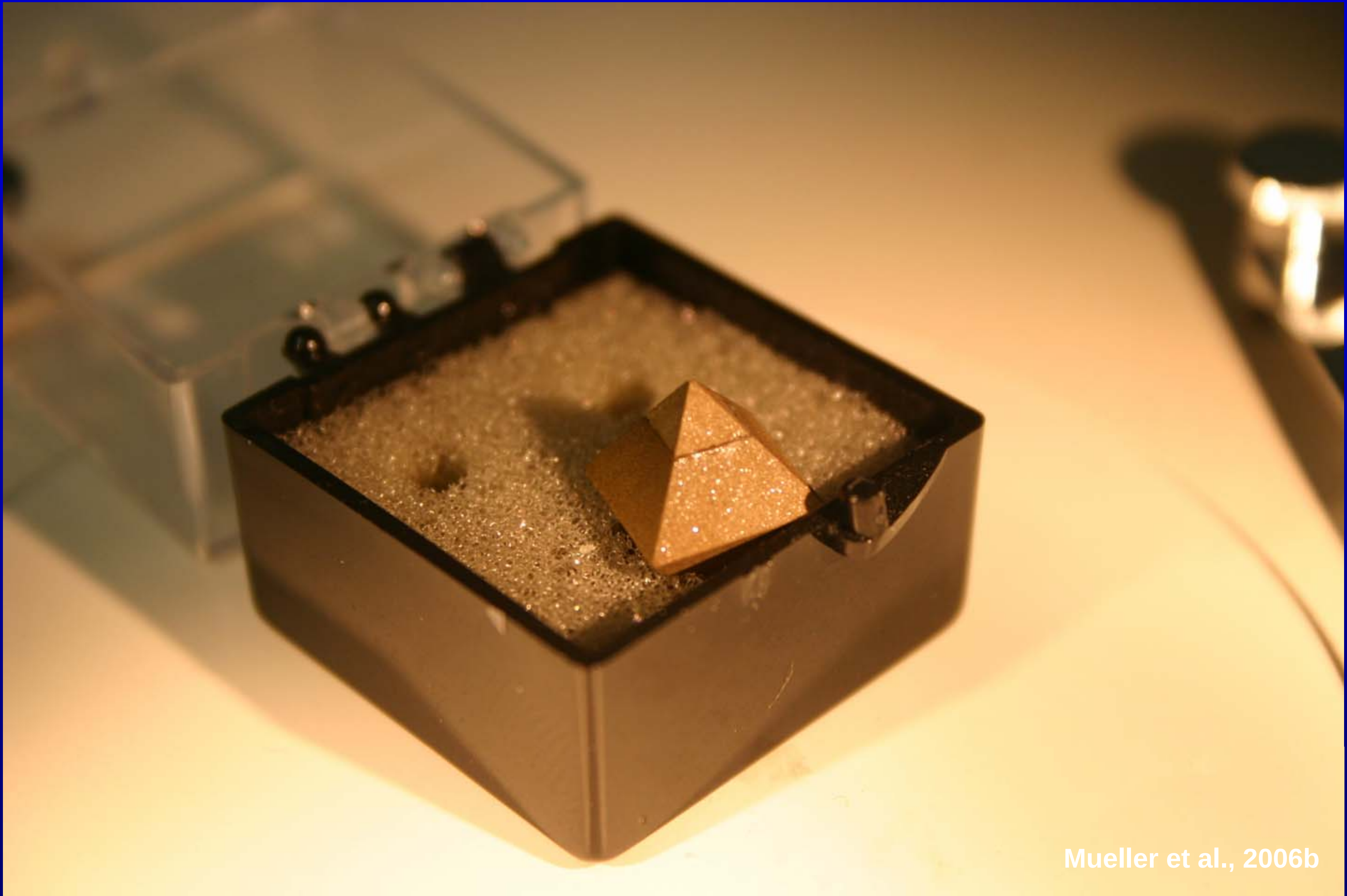
GFZ POTSDAM
Hans-Joachim.Mueller@gfz-potsdam.de

opposed anvil set-up in second-stage octahedron



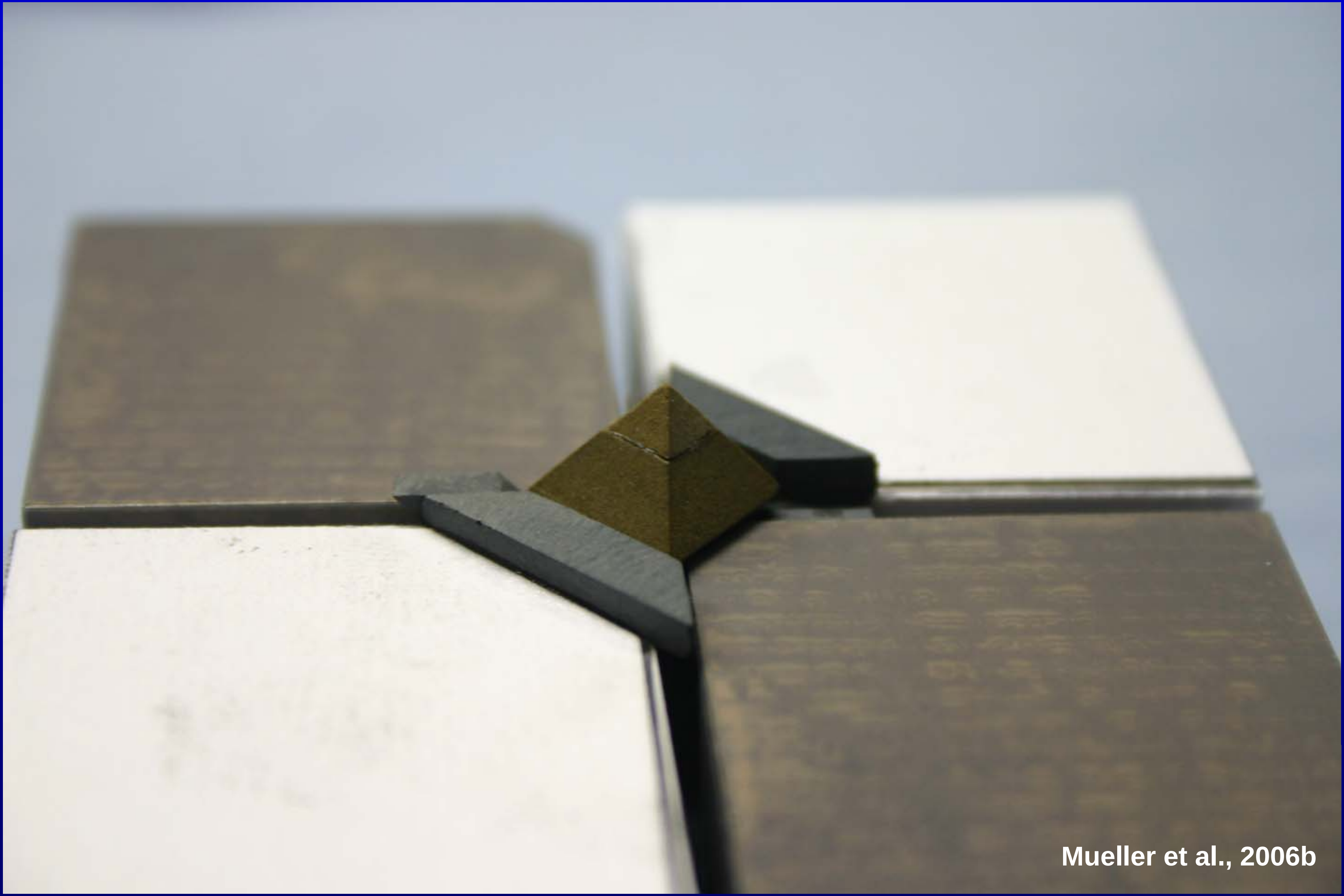
Hans-Joachim.Mueller@gfz-potsdam.de

Mueller et al., 2006a



MAX200x – triple-stage octahedron

GFZ
POTSDAM
Hans-Joachim.Mueller@gfz-potsdam.de

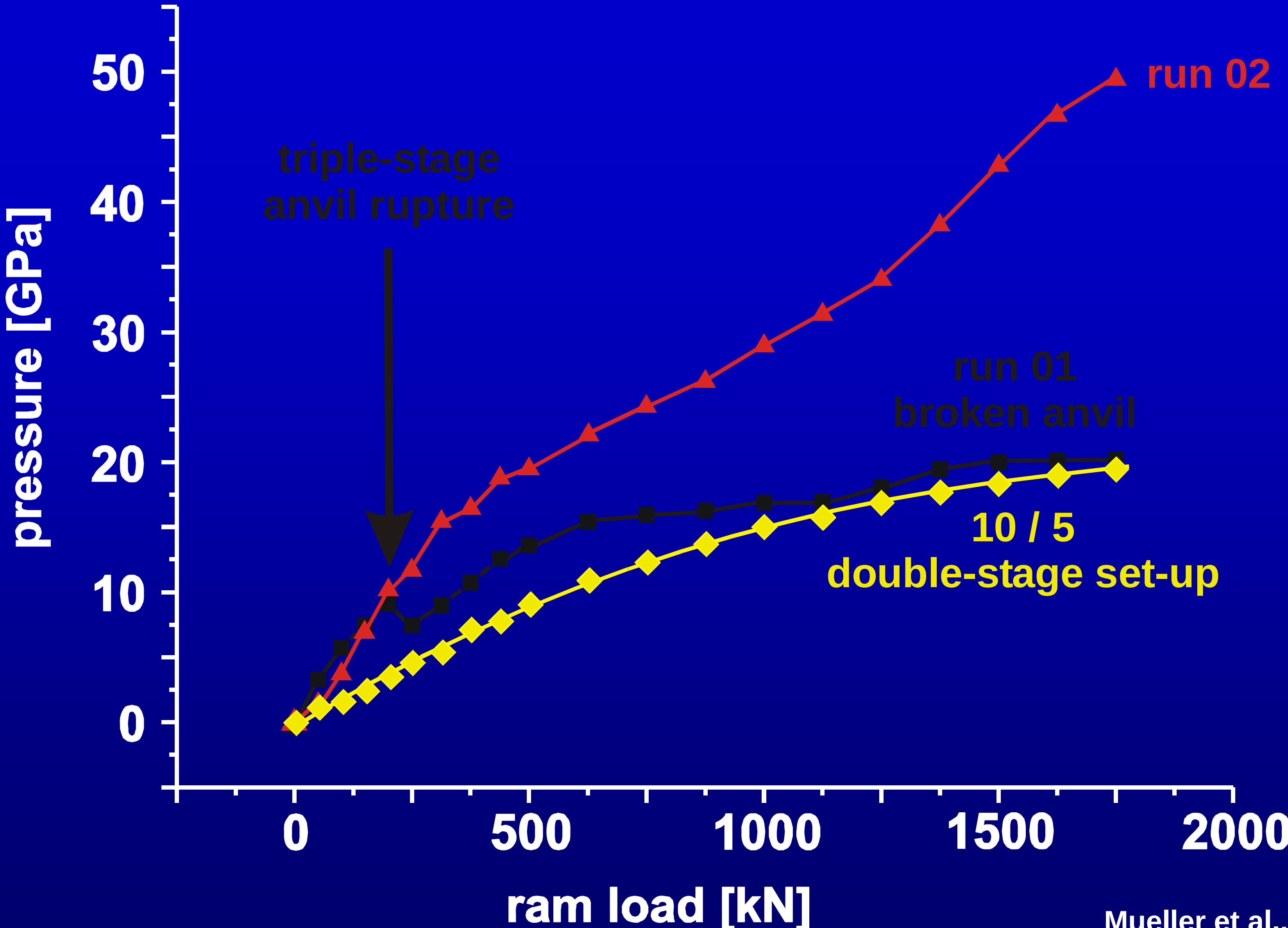


MAX200x – triple-stage set-up in preparation

Hans-Joachim.Mueller@gfz-potsdam.de

MAX200x – triple-stage set-up after the hp-run

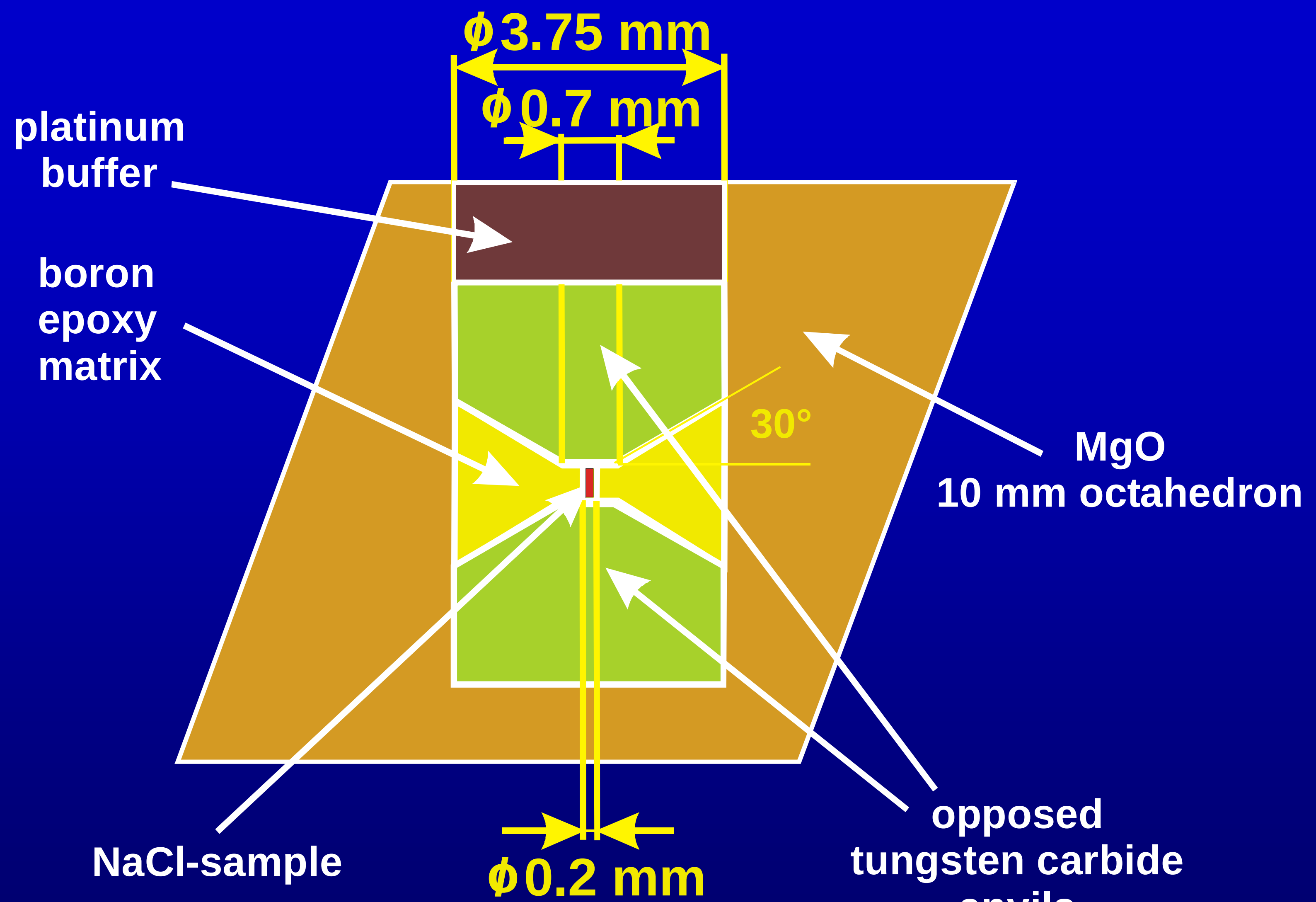
Hans-Joachim.Mueller@gfz-potsdam.de



Mueller et al., 2006b

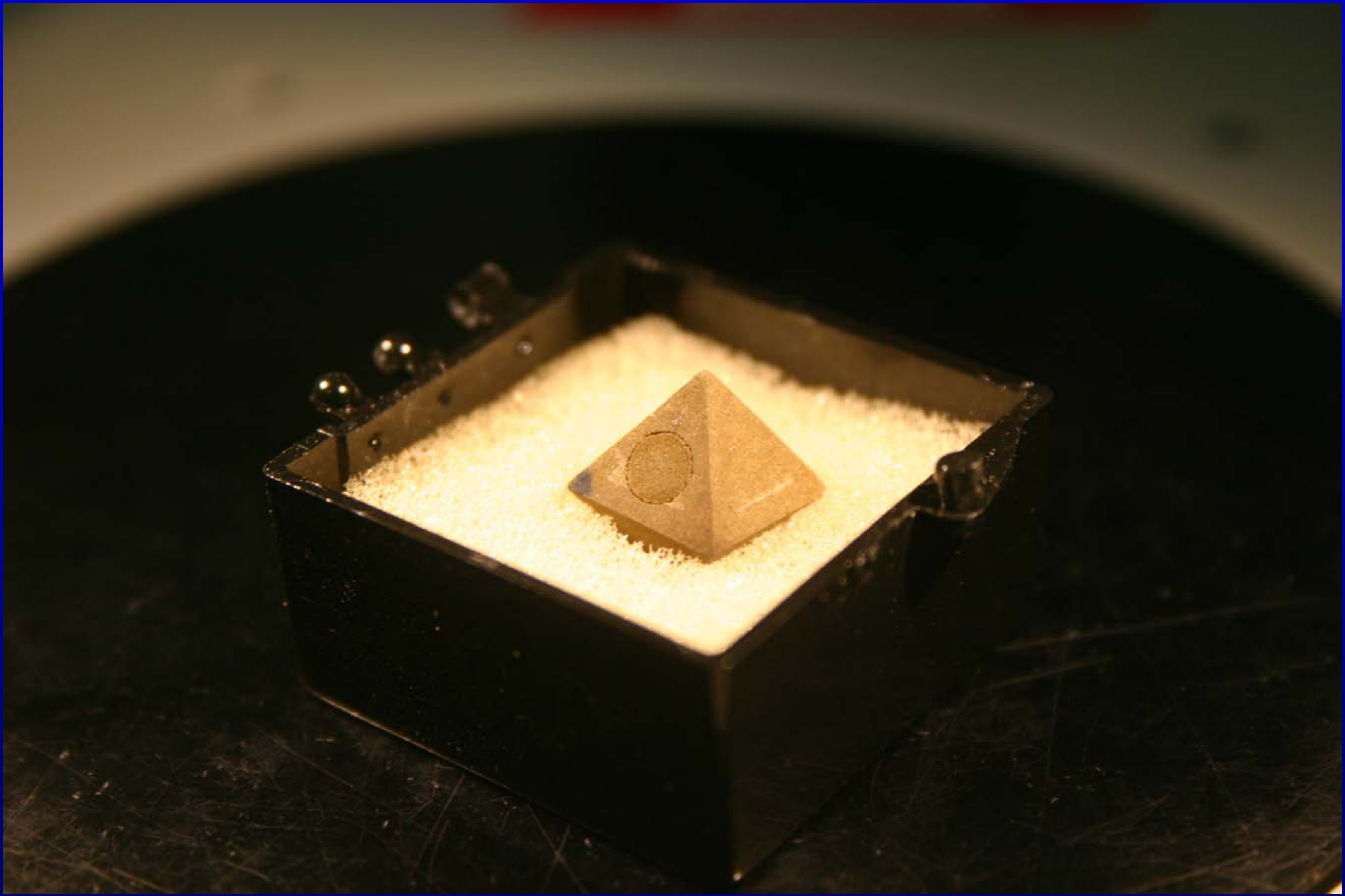
MAX200x – pressure calibration of triple-stage set-up

GFZ POTSDAM
Hans-Joachim.Mueller@gfz-potsdam.de



opposed anvils ultrasonic set-up

Hans-Joachim.Mueller@gfz-potsdam.de

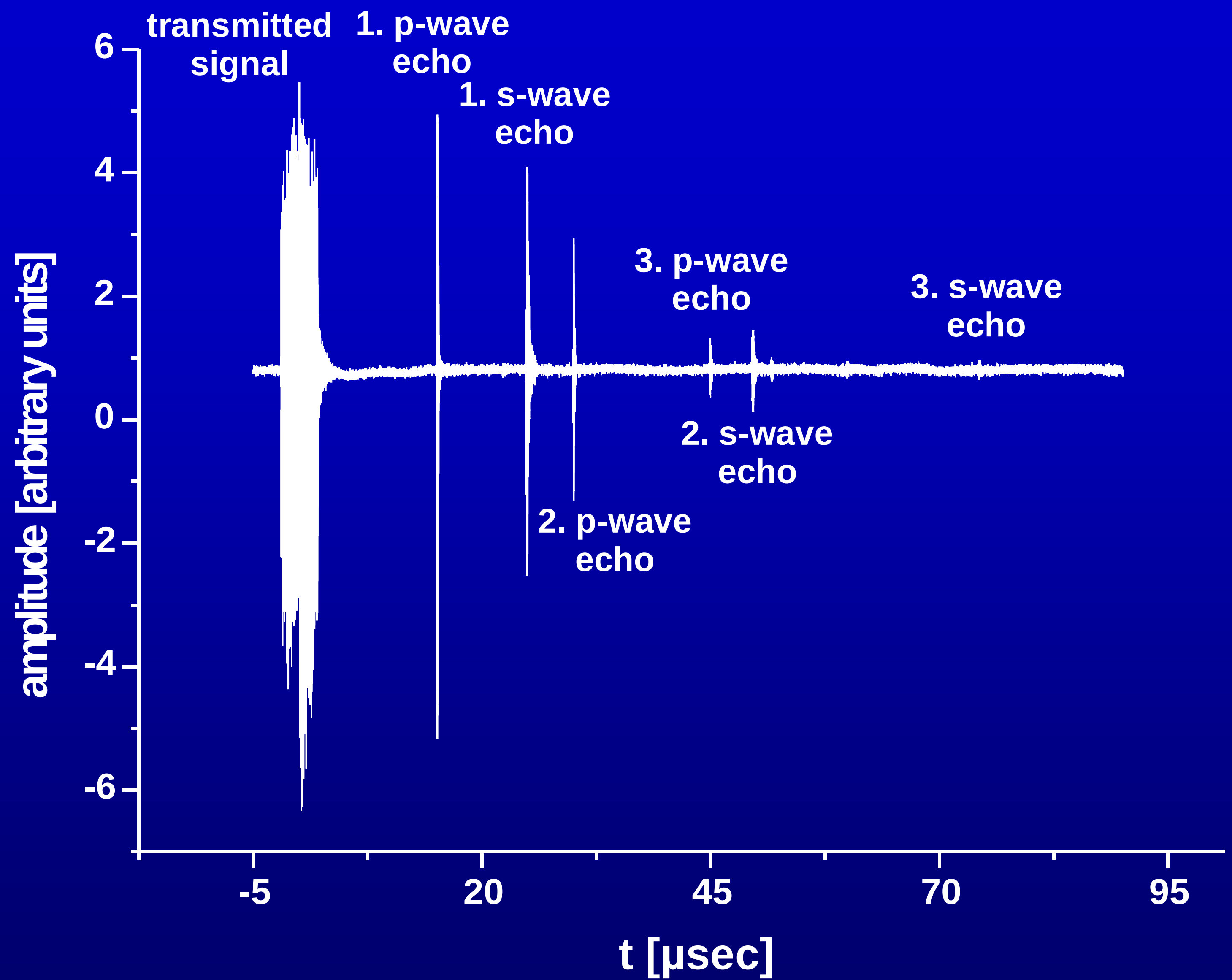


opposed anvils ultrasonic set-up

Hans-Joachim.Mueller@gfz-potsdam.de

MAX200x – Ultrasonic set-up with triple-mode transducer

GFZ POTSDAM
Hans-Joachim.Mueller@gfz-potsdam.de



MAX200x – DTF of a triple-stage set-up

Hans-Joachim.Mueller@gfz-potsdam.de

summary

- multi-staging – as track to higher pressures in LVP

- ultrasonic interferometry – DTF technique

- ultrasonic opposed anvils set-up

- X-radiography – sample length measurement

- standard-free pressure measurement in multi-staging

- triple-stage multi-anvil techniques

- further development for face to face triple-staging needed

Hans-Joachim.Mueller@gfz-potsdam.de


Thank you
for your
attention!
Iceland, September 2006