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Two-stage multi-anvil apparatus in measurement of low-temperature

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ABSTRACT: *The two-stage 6-8 multi-iron block (MA8) mechanical assembly is a significant huge volume, high-pressure strategy that has been generally utilized in the high pressing factor mineralogy and material union, fundamentally at room temperature or above. As of late, we have effectively built up a two-stage MA8 device for low-temperature actual property estimations. The principal stage iron blocks at top and base sides are manufactured as a solitary piece to re-duce the complete size of the tube shaped module, which is placed in a top-stacking high pressing factor cryostat and compacted by a 1000 ton water driven press. A castable, split octahedral gas-ket with incorporated balance was explicitly planned to present the electrical leads from within test holder loaded up with a fluid pressing factor communicating medium. By utilizing tungsten carbide (WC) second-stage solid shapes with a shortened edge length of 3 mm and an octahedral gasket with an edge length of 6 mm, we have effectively produced pressure more than 20 GPa at room temperature. Since the high pressing factor breaking point can be pushed to almost 100 GPa by utilizing the sintered precious stone second-stage 3D squares, our MA8 device has an extraordinary potential to extend the current pressing factor limit with regards to exact low-temperature estimations with an enormous example volume.*

I. INTRODUCTION

Pressure is a fundamental parameter like temper- ature that governs the states of matter. The ap- plication of high pressure can induce structural or electronic phase transitions or precisely tune the structural and physical properties. In condensed matter physics, the combination of high-pressure and low-temperature environments provides avery fertilegroundforexploringnovelquantumstatesof matter and exotic phenomena. For example,pressure can induce a magnetic quantum critical point, near which the Landau Fermi-liquid behavior usu- allybreaksdownandunconventionalsuperconduc- tivity frequently takes place due to the presence of strong quantum fluctuations. Therefore, it is im- portant to develop a high pressure apparatus for low-temperaturemeasurements.

Despite the sophisticated low-temperature tech- nologiesexistent,thehigh-pressuredevicesusedin low-temperature conditions remain to be further developedduetothespaceconstrainandotherspe- cific requirements, such as pressure homogeneity, sample volume, etc. Currently, piston-cylinder cell (PCC)[1,2]anddiamondanvilcell(DAC)[3,4]are two widely used commercial high-pressuredevices for in-situ physical property measurements at low temperatures. PCC offers a large sample spaceand relativelygoodhydrostaticitybyemployingaliquidpressure transmitting medium (PTM) [5], but the maximum pressure is usually limited to 4 GPa [1], whichisinsufficientformanystudiesincondensed matter. Although the DAC [3] can achieve ultrahighpressuresandalloweasyaccessfortheelectromagneticradiations,thetinysamplespacemakesit difficultforin-situphysicalpropertymeasurements requiring electrical contacts, and the solid PTM usually employed renders severe non-hydrostatic pressureconditions.

Besides the PTM, the level of pressure hydro- staticity/homogeneity also depends on the com- pression geometry. In comparison with DAC, multianvil-type (MA) apparatus can maintain bet- ter pressure homogeneity even if the PTM becomes solidified at low temperature and/or high pressure [6]. In addition, the MA apparatus can reach pres- sure above 10 GPa, much higher than PCC. The single-stage cubic anvil cell (CAC) device devel-oped in the Institute for Solid State Physics, the University of Tokyo (ISSP, UT) [7] is one typi- cal MA apparatus that can generate hydrostatic pressures up to 15 GPa. The design of miniature “palm”-type CAC also enabled integration with ^{3}He or dilution refrigerator so as to reach temper- atures as low as 10 mK [8, 9]. These developments of cubic-type apparatus were essential for us to dis- cover novel quantum phenomena [10] and new su- perconducting materials [11] recent years.

To pursue more exotic phenomena in an ex- tended pressure range, there is always a demand forthedevelopmentofdevicesreachingevenhigher pressures. In this regard, the two-stage 6-8 multi- anvil (MA8) apparatus originally developed in 1970s by Kawai and Endo becomes an excellent option [12]. In this case, the first stage of six anvilssurroundsacubiccavity,inwhichitisplaced the second stage, consisting of eight cubes with truncated corners forming an octahedron. After 40 years of developments, the MA8 apparatus has gain great success and has been widely used in high-pressure mineralogy and synthesis of mate- rials. Depending on the strength of the second- stage anvils, the maximum pressure of MA8 are used for high-pressure studies at or above room temperature. In this paper, we report the devel- opment of a two-stage MA8 apparatus for precise low-temperature physical property measurements in ISSP,UT.

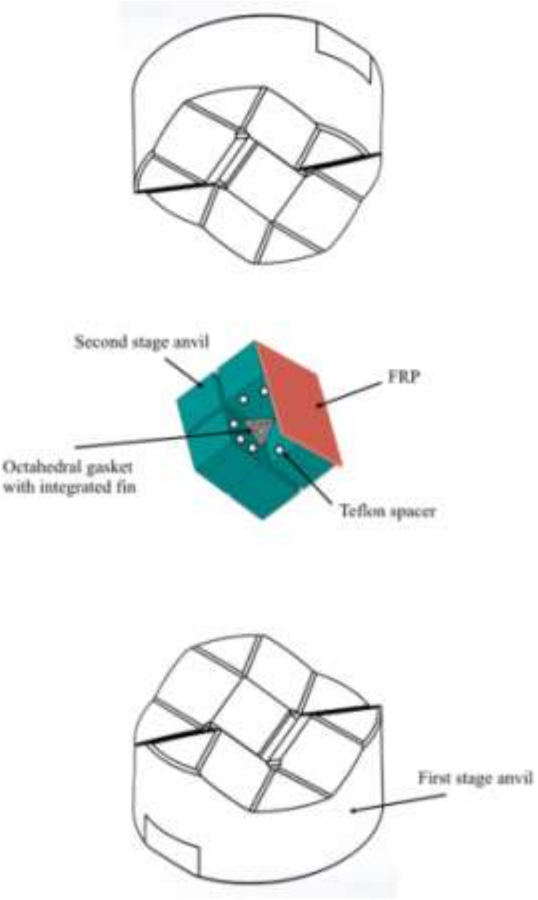

Figure1: Schematicillustrationofthefirst-stageand second-stageanvils.

II. EXPERIMENTAL SETUP AND RESULTS

i. Two-stage MA8device

For the commonly used two-stage MA8 appara- tus, the first-stage six anvils (three on the top and three on the bottom) made of hardened steel are usually built into a thick-wall steel ring (Kawai type)orcontainedinaremovablecylindricalmod- ule (Walker type) [13]. Such designs are not suit- able for low-temperature applications because the whole MA8 device has to be inserted into a cryo- stat.ToreducethetotalsizeoftheMA8device,we designedthefirst-stage three anvilsontop and bot- tom sides as a whole piece, as shown inFig. 1. We have also used a nonmagnetic NiCrAl alloy tofab-

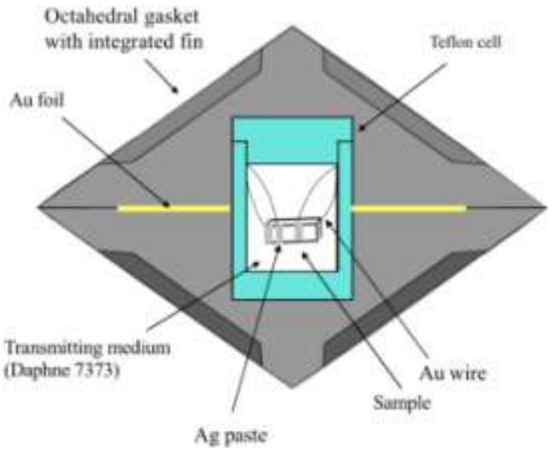

Figure2:Cross-sectionalviewoftheinternalconfigu- ration of the gasket with tefloncell.

ricatethepairofcylindricalfirst-stageanvilsinor- der to apply magnetic fields. The first-stage anvils haveanouterdiameterof154mmandformacubic cavity with edge length of 32.3 mm. The second- stage anvils, consisting of eight cubes with trun-cated corners, are similar to the commonly used MA8 apparatus. Here, we employed nonmagnetic WC (TMS05/MF10 grade from Fujilloy) with an edge length of 18 mm and truncated corner of 3 mm. As a common practice, these WC cubes are held together with six pieces of Fiber-Reinforced Plastics (FRP) pads, which are 0.5 mm in thick- ness and 36 36 mm in area. These FRP pads also serve as an insulation to the first-stage anvils. The insidesurfacesofthesecond-stagecubesarepasted with three 1.0 mm cubic Teflon spacers to prevent electrical contact with adjacentanvils.

ii. Gasket design and sampleassembly

TheadoptionofaliquidPTMisessentialtomain- tain a relatively good pressure homogeneity.How- ever, the conventional design of octahedral gasket and sample assembly used for the MA8 apparatus also need to be modified in order to accommodate a sample container filled with liquid PTM. Forthis purpose, we adopt the castable, split octahedral gasket with integrated fin, which are made fromCeramacast 584-P and Ceramacast 584-L(100:28

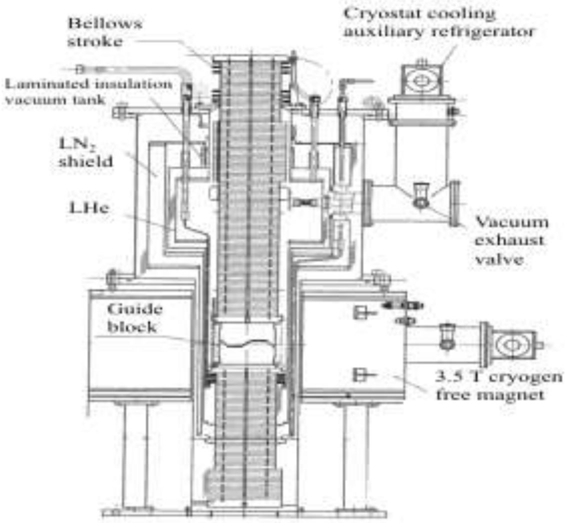

Figure3: Cross-sectionalviewofthetop-loadingcryo- stat.

weight-in-weight) potting compound fromAremco Products, Inc. The half-octahedral gaskets with in- tegrated fins are made in-house in our laboratory according to the procedures described in Ref.[14]. Theedgelengthoftheoctahedronis6mmandthe thickness of the gasket fin is 1 mm. Figure 2 de- picts the internal configuration of the gasket with the sample hanging inside the

Teflon capsule(I.D.1.5mm,O.D.2.0mmandlength2.5mm),whichis the same setup used in the cubic anvil cell[7]. The Teflon cell can be filled with a liquid PTM suchas Daphne 7373 or Glycerol, and the electrical leads are introduced via gold foil to the surfaces ofocta- hedral gasket, which in turn contact with the WCcubes.

iii. Top loading high-pressurecryostat

Figure 3 shows a schematic cross-sectional view ofthetop-loadinghigh-pressurecryostat,inwhich the MA8deviceisplacedinbetweentheupperand lower pushing columns. Details about the design of the high-pressure cryostat can be found in an earlier publication about the cubic anvil cell apparatus [7]. The low-temperature condition (down to 2 K) is realized by filling the cryostat with liquidnitrogenandthenheliumwithproperpumping. Precise temperature control between 2 and 300 K was achieved by attaching a resistance heater onto the MA8 device. The pressure is generated by using a1000-tonhydraulicpress,whichcanmaintain a constant loading force over the MA8 deviceover the whole temperature range. In addition, a 3.5 Tesla helium-free superconducting magnet with a large bore size is also installed and the center of themagneticfieldisalignedwiththesampleinthe MA8device.

Table1: Phasetransitionsaspressurecalibrants[15].

Sample	Pressure (GPa)
Bi	2.55, 2.7, 7.7
Sn	9.4
Pb	13.4
ZnS	15.6
GaAs	18.3

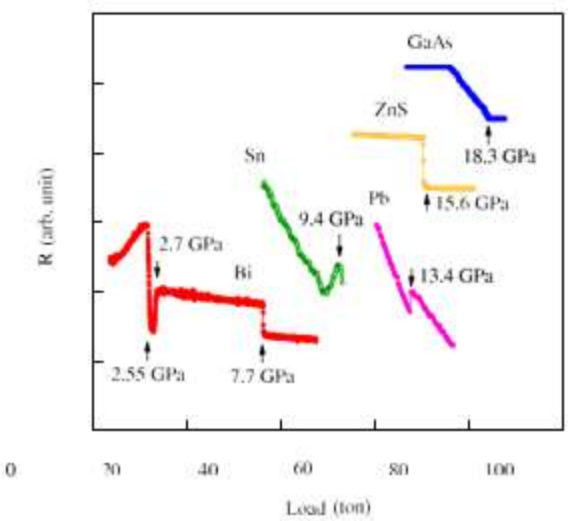

Figure4: ElectricalResistanceofBi,Sn,Pb,ZnSand GaAs as a function of loadingforce.

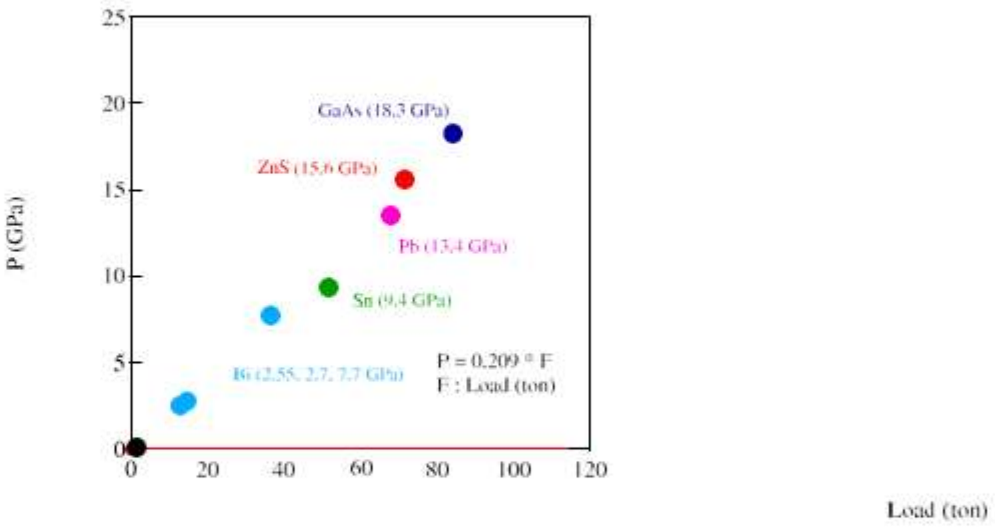

Figure 5: Pressure calibration line for a two-stage multi-anvil high pressure cell.

iv. Pressurecalibration

We have performed fixed point pressure calibra- tion at room temperature by detecting the charac- teristic phase transitions of Bi, Sn, Pb, ZnS and GaAs in electrical resistance. A standard four- probe method was used to measure the resistance of each sample. Table 1 summarizes the transition pressure of these materials from previous studies [15].

Figure4showstheelectricalresistanceofBi,Sn, Pb, ZnS and GaAs as a function of loading force at room temperature. As can be seen, the char- acteristicphasetransitionsofBiat2.55,2.7and 7.7 GPa were clearly observed at loading force of 12.2, 13.7, and 36.2 tons, respectively. We defined the phase transitions which are the offset. Simi- larly, the resistance anomalies of Sn and Pb at 9.4 and 13.4 GPa were also observed at 51.7 and 67.9 tons, respectively. In addition, the metallization of ZnS and GaAs at 15.6 and 18.3 GPa weresuccess- fully observed at a loading force of 71.1 and 83.9 tons, respectively. Although the employedDaphne 7373 PTM becomes solid at about 2.3 GPa, these characteristic phase transitions remain very sharp, signaling an excellent pressure homogeneity up to at least 20 GPa due to the multi-anvilgeometry.

Based on these measurements, we have plottedinFig.5thepressurecalibrationcurveforourtwo-stageMA8apparatusinstalledwithWCcubeshav- ing a truncated corner of 3 mm. As can be seen,allthecalibrationpointsfallnicelyonalinercurve described by P (GPa)= 0.209 Force (ton). From the extrapolation, we can reach about 25 GPa ata loading force of 120 tons, which is a muchlower force than those reported in the literature employ- ing MgO octahedron plus extra pyrophyllite gas-kets. In the latter case, a large portion of load- ingforcewasdissipatedontherelativelysoftpyro- phyllitegasketsothatthecalibrationcurveusually tends to saturate at higher loading forces. In con- trast,themuchimprovedpressureefficiencyinour MA8 apparatus should be attributed to the octa- hedral gasket with integrated fin, which is muchharder than pyrophyllite. As mentioned above, the maximum pressure at which MA8 can be pushedto is over 40 GPa by using a tapered second-stage WCanvils[16],ortonearly100GPabyemploying much harder sintered diamond cubes [17]. It can be thus foreseen that the pressure capacity of our MA8 apparatus can be furtherimproved.

III. CONCLUSIONS

We have successfully developed a two-stage6-8 multi-anvil apparatus for accurate high-pressure and low-temperature measurements. By using tungsten carbide second-stage cubes with trun- catedcornersof3mmandcastableoctahedralgas- ket with an edge length of 6 mm, we can generate pressures over 20 GPa at a relatively low loading force of 100 ton. An excellent pressurehomogene- ity/hydrostaticity up to 20 GPa has been demon- strated in our MA8 apparatus, which is expected to reach even higher pressures by employing WC anvils with smaller truncation sizes or sintered di- amondanvils.

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