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A Brand-New 6-6 Assembly for a Cubic-type of Multianvil Apparatus

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We developed a new way of assembling a second stage, which includes a frame bundling a set of second-stage anvils when loading a high-pressure cell using a DIA-6 type of multianvil apparatus. A newly developed tool can assemble a whole set within 4 minutes with a precision required for high-pressure experiments.

1 Introduction

Nowadays, science under extremely high pressures beyond 100 GPa is mainly developed using a diamond anvil cell. However, a multianvil apparatus has still been a powerful tool with which to generate high pressures in material and Earth sciences and technology. Here, we focus on a cubic-type apparatus, often referred to as a multianvil-6 (MA-6), consisting of six anvils. MA-6s have been installed in not only laboratories but also synchrotron x-ray and neutron beam facilities worldwide to carry out in situ experiments. For example, MAX-80 and MAX-III installed in beamlines NE5C and NE7A, respectively, are of this type. The mechanism of generating high pressure is simple; the external load exerted on the main ram is transmitted to the anvils equipped with an anvil guide of an MA-6. The anvils then push a sample embedded in a cubic pressure-transmitting medium. Here, we call such a traditional method one-stage compression.

Instead of this one-stage compression, many apparatuses have begun to adopt a two-stage compression method since around 2010[1,2]. The outer set of anvils compresses the six small anvils bundled with a frame. The second-stage assembly (SSA), consisting of the set of inner anvils holding a sample cell, is removable upon exchanging a sample. The portability of the SSA facilitates replacing the anvils by those with a different truncation edge length (TEL), depending on a pressure range desired, thus taking optimal advantage of the beamtime available when a user plans to use sets of anvils with different TELs.

However, when users perform their experiments using a set of inner anvils of the same TEL, a two-stage compression is not necessarily efficient; it instead produces extra loss time much unless more than one SSA is available. An SSA is composed of a set of inner anvils, stuck together by Teflon™ chips of a particular size, which is held in a metal frame wrapped up by a Kapton® tape to ensure electric insulation. A set of chips with appropriate height, such as pieces of balsa sheets with a specific thickness, must be stuck on the corners of the frame to center exactly the SSA (see Fig. 1 left panel). The Teflon chips and the balsa pieces are required to make the virtual center of the inner anvils coincide with that of the outer anvils. We call this adjustment ‘centering’ here. A situation in which preparing the SSA cannot proceed in parallel to the experiment results in a severe loss of efficiency of the whole experiment. We should mention that purchasing these specific parts and a set of inner anvils is burdens on the user side.

Our project has started to address these issues and set the following goal: A user alone can set up the SSA and restart-up compression within 15 min upon a sample change. Although this time will be much less than a usual time duration of more than 1 hour when the conventional assembling method is adopted, we will aim the same degree for the accuracy of centering as attained by the ordinary method.

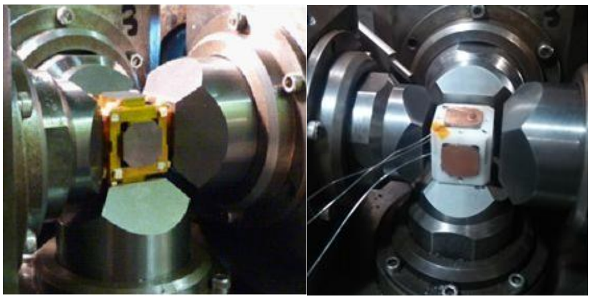

Fig. 1: The SSA mounted in the first-stage outer anvils of MAX-80 installed in BL NE5C, KEK-AR. It takes more than one hour to assemble a conventional SSA, i.e., an electrically insulated metallic frame bundling inner anvils, which support a sample cell (left). In contrast, the new assembly consisting of a POM frame (right) allows a single user to finish setting within 4 min using the unique tool devised.

2 New Development

To this end, we first paid attention to the roles of a frame supporting a set of inner anvils: (i) It must help the inner anvils to ensure the precise centering. Most of the users use a metallic frame because of the excellent workability of metals. (ii) Therefore, a frame must be electrically insulated for electric current to flow through a heater enclosing the sample (container) because we usually measure samples at high temperatures under pressure.

The condition (ii) is satisfied if we use insulators as a frame at the outset. Indeed, Dr. Kikegawa of IMSS, KEK, has used a plastic frame made of PEEK to bundle the inner anvils. According to his suggestion, we first replaced a metal frame by a PEEK frame. Nevertheless, at the same time, we started to look for other plastic materials, which have similar workability but are less expensive. Finally, we encountered POM, which can offer comparative performance though the melting point is about half as high as that of PEEK. To meet the condition (i), we devised a unique tool with which to attain the centering within 4 min with the required precision. The anvils are finally clamped with six small screws to the frame. Such works as shielding a frame with Kapton® tape, adjusting the position of a frame with balsa pieces, and joining the anvils with Teflon™ chips are now no longer required (Fig. 1 right panel). We are now free from such a laboring job and the extra cost.

The development was accelerated since 2018 and finished at the end of FY2019. The Ehime Industrial Promotion Foundation supported us financially during the period.

3 Verification Experiment

We examined for plastic frames made of PEEK and POM a series of necessary tests such as the extent of deformation before and after holding them under 40 degrees centigrade, which is the expected temperature they experience when placed in an MA-6 on actual experiments. We confirmed no quantitative or qualitative changes in the plastic frames against those tests.

We applied for the experiment at KEK because we need to check our SSA can be used safely in high-pressure experiments. Besides, we have to confirm in situ that the centering is satisfied with the desired accuracy.

We conducted verification experiments using MAX-80 installed at NE5C from 2018 through 2019. Four opportunities were given to the experiments. We used TEL of 4, 6, and 8 mm as the internal anvils. A W-Re wire piece of diameter 0.125 mm and length 1 mm was used as a dummy sample. A typical compression and decompression cycle are as follows: We first applied 30 tons of load at room temperature and then heated a dummy sample to 1000 or 1500 K. We loaded up to 100 or 120 tons at that temperature. We measured pressure at the sample position using a pressure standard, NaCl, at every addition of 20 tons of load. We also took a photo of the transmission image of the dummy sample at those pressures. The diameter of the sample was almost comparable to the width of a gap between the anvils at the highest load applied when using anvils of TEL 4 mm.

3 Results and Discussion

The temperature of the frame was monitored during each experiment. While the sample was held at 1500 K, a PEEK frame position never exceeded forty degrees centigrade. In the same situation, the temperature of a POM frame stayed even several degrees lower.

We are planning to make a detailed report elsewhere on the results of the verification experiment, including such a temperature history, together with the precise design of the assembling tool we developed [3]. We only show here in a visually appealing fashion that the tool can bring about satisfactory centering.

As an example, we show in Fig. 2 a situation we observed when we compressed the sample using a set of inner anvils of TEL 6 mm. We can easily confirm that a dummy sample (a bit of wire) always centers the anvil gap until the highest load applied. The situation remained the same even when the conditions became severer, i.e., when the inner anvils of TEL 4 mm were employed.

Fig. 2: A transmission image of the dummy sample in an anvil gap when a POM frame was employed is shown. (a) Anvil gap 2.11 mm at ambient conditions, (b) 0.99 mm at 5.0 GPa and room temperature, (c) 0.94 mm at 3.0 GPa and 1500 K, and (d) 0.78 mm at 5.8 GPa and 1500 K.

We could thus reach our goal held up when this R&D project has started. However, we could uncover a problem within a series of verification experiments, in which we experienced blowouts. We found that a blowout at a high temperature altered the surface conditions of a POM frame. This alteration makes the slip characteristics of the frame change inhomogeneously. Therefore, even though the size of a frame is unaffected by a blowup, and it holds the inner anvils at the right position, the anvils would not slide uniformly upon loading. This unsynchronized movement of the inner anvils would cause a rotation of a sample cell, thereby making beams miss the sample. We confirmed a similar alteration of a PEEK frame, which experienced a blowout under elevated temperatures. Resolving the issue is our next concern.

4 Summary

We confirmed that our newly designed POM frame, which is less expensive than a PEEK frame, can safely and stably hold a sample at high temperatures and pressure. The assembly tool devised allows us to quickly (within 4 min) center the sample with the desired precision. However, we cannot recommend reusing a POM (PEEK) frame if it experiences a blowout.

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References

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Research Achievements

For both the frame and assembling kit, we shall not apply for a patent nor for a design right, which may prevent high-pressure science from extending its reach. Shinkou Kouki Co. has just released a whole set of tools at a reasonable price.

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